THE JOURNAL

OF THE

AMERICAN LEATHER CHEMISTS ASSOCIATION

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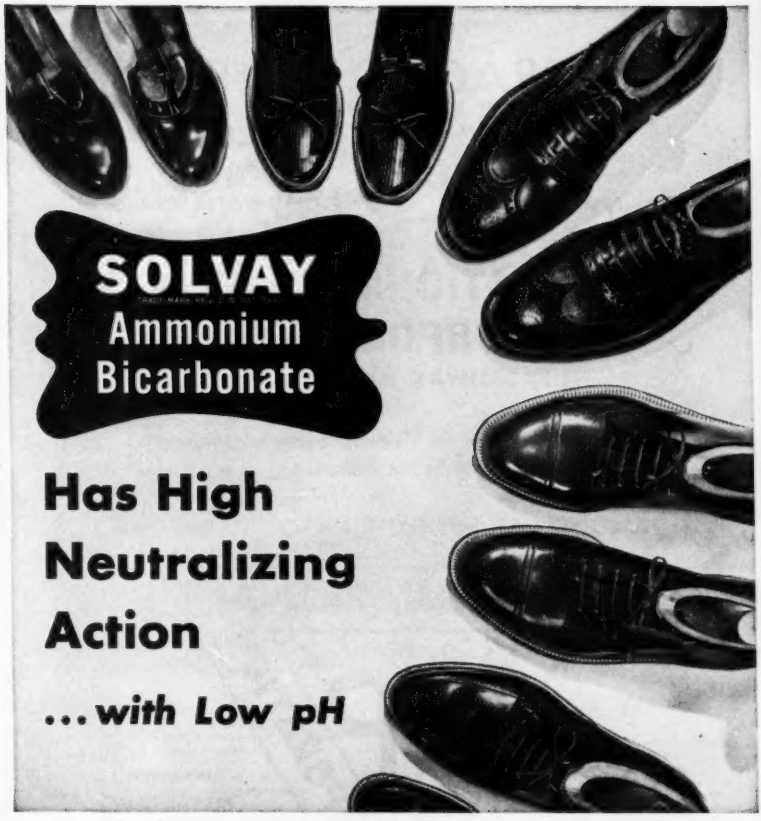

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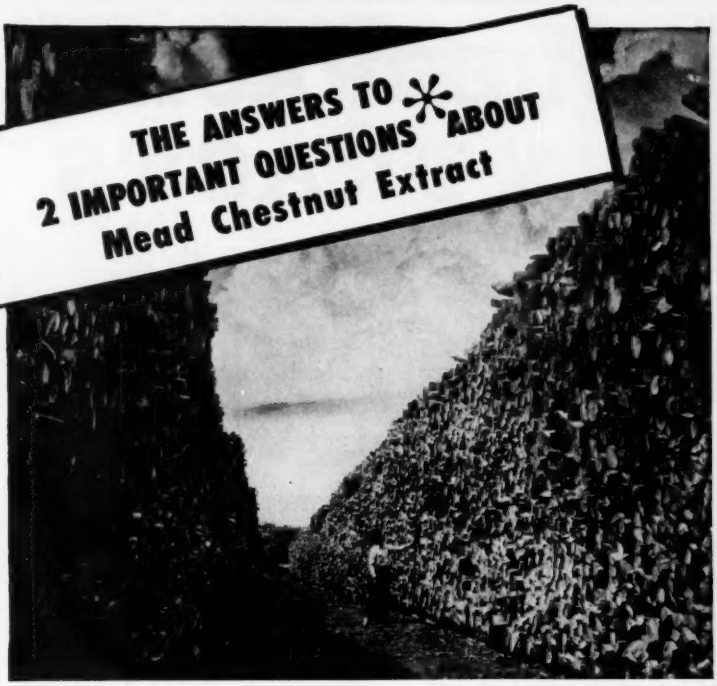
THE ANSWERS TO 2 IMPORTANT QUESTIONS* ABOUT Mead Chestnut Extract



This is only a portion of the many stockpiles of chestnut logs that insure a continuous supply of Chestnut Extract.

*** 1. Is the supply of Mead Chestnut Extract adequate?**

Decidedly yes. Recent surveys show that there is enough chestnut wood in our forests to serve the leather industry for many years to come. Huge chestnut stockpiles, such as the one shown above, are converted under chemical control at five extract plants, insuring a steady supply of Mead Chestnut Extract.

*** 2. Why is Mead Chestnut Extract preferred over other tanning agents?**

Mead Chestnut Extract is a superior tanning material, made by America's largest producer of vegetable tanning extracts. Repeated experience has shown:

A. Chestnut tanned leathers do not oxidize, darken on exposure to light, or develop an uneven color on aging, as do leathers tanned with many other materials, vegetable or synthetic.

B. Since the tanning system demands acid, the use of chestnut, with its plumping acidity, saves you money.

C. Mead Chestnut Extract eliminates up to 28% of leachhouse losses.

D. There are no hidden costs. When you use Mead Chestnut Extract, no time- and money-consuming preparation is required.

Ask about Mead Cold Soluble Chestnut Extract for retanned leathers. And—for more pounds of plumper leather—standardize on Mead Chestnut Extract.

IT'S A FACT!

Mead Chestnut Extract will produce more pounds of plump leather per unit of tannin than any other tanning material.

Vegetable Tannin	Tan Yard Yield (*white weight basis*)
Chestnut Wood Extract	67%
Tanning Material A	61%
Tanning Material B	63%

Calculate your tanning material costs on a leather basis as well as on a tan unit basis. Mead Chestnut Extract, producing four to six pounds more leather, means positive savings.

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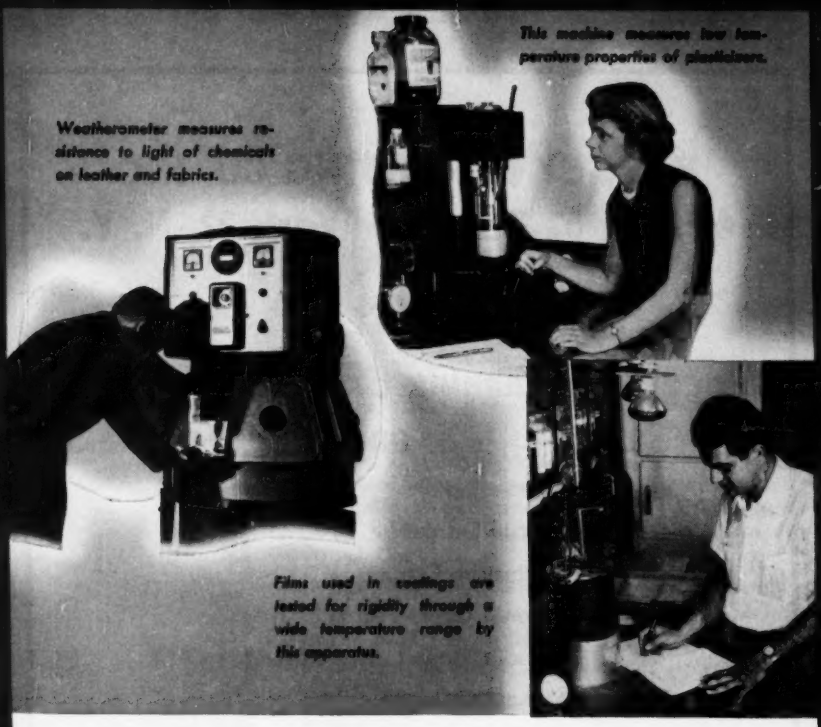
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A case in point is ZIRCOTAN, the zirconium tanning agent for producing through-white leather. Initially our interest in zirconium compounds centered around their use as opacifiers for porcelain enamel. Subsequent work on the chemistry of zirconium led to the development of ZIRCOTAN.

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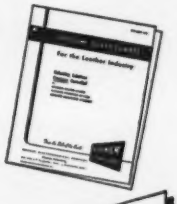
Hooker Bulletin 500 tells more about Sulfhydrate Unhairing Solutions. It suggests procedures for calfskins, hides for sole leathers, for side and upper leathers, and for specialties. A copy will be sent you free when requested on your company letterhead.



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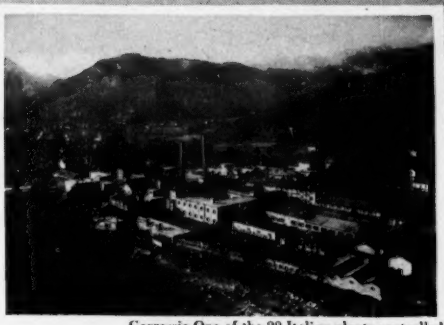

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VOL. XLIX JANUARY 1954 No. 1

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Members Note:

Article V - 2, Nominations (c)

Additional candidates may be nominated by petitions, signed by 15 or more active members. Such petitions shall reach the Secretary not later than April 1st, and shall contain a statement of the willingness of the nominee to serve if elected. No member shall sign more than one petition for each office to be filled.

COUNCIL MEETING MINUTES

November 10, 1953
University of Cincinnati
Cincinnati, Ohio

The meeting was called to order at 10:00 A. M. by President Turley. Present were Messrs. Flinn, Collins, Ross, Wilson, Henrich, Williams, Teas, Meo and O'Flaherty.

The minutes of the two Council Meetings held in June were given a final approval.

President-Elect Flinn reported as Coordinator of the Technical Committees.

The chairmen of the technical committees met at the time of the Annual Meeting and each has reported on the scope of the work for 1952 and 1953. These reports are now being made into one report and will be published in the January Journal.

The new Mechanical Leather Committee has become organized and adopted a program under the chairmanship of Mr. Berens.

The Leather Finish Committee chairman has requested that he be released from chairmanship due to other duties. The President is to appoint a new chairman.

Standards and Specification Committee has functioned well and has submitted a report.

Preparatory and Post Tannage Committee has submitted revised methods on Oil and Greases and Beamhouse Methods. These will be edited and published.

It was suggested by Council that Method No. 3 for Nitrogen Determination, using ½ gram sample, be considered as a referee method.

Tannery Practice Committee reported no action but the sub-committee of Industrial Waste Disposal is active and one member is preparing a paper on waste disposal for tanneries.

The Physical and Mechanical Property Committee under Mr. Maeser has submitted an additional method – Fire Resistance – this will be published in the January Journal. All Physical Methods will be republished in one group for distribution.

The Mineral Tannage Committee presented a report and the chairman, Dr. Seligsberger, has asked to be released and a new chairman appointed.

The Vegetable Leather Committee has reported that the sub-committee on Hide Powder is now examining a new lot of hide powder and it will shortly be released.

The Tannin Analysis Sub-Committee has submitted their methods in revised form. They are to be published in the January Journal.

Mr. Rogers has requested authority for the committee to now revise the Method on Vegetable Leather Analysis. Council heartily approves this.

Dr. Flinn then reported on the Methods booklet.

It was agreed by the Editorial Committee that the general plan of format, numbering and paragraphing as used in KK-L-311 is to be followed. The numbers of the paragraphs and sub-paragraphs are to be extended out from the body of the text in a column in front of the text.

The Physical Testing Methods are to be so arranged and published for distribution.

The Tannin Analyses Methods of Vegetable Leathers are then next to be published.

The Methods on Oils and the Beamhouse Methods have been returned to Dr. Merrill for a final review and approval.

The other methods are to be published as they are revised and put into proper format.

Because of the past delay, a motion by Mr. Harry Wilson, seconded by Mr. Leslie Collins, and unanimously approved by Council, a sum up to $500.00 is to be made available to the Technical Committee Coordinator to pay for clerical help in getting the methods ready for publication.

Mr. John Teas then reported for the Ways and Means Committee.

The matter of the Directory cost was discussed and it was approved by Council that advertising space in the 1954 Directory would be $10.00.

The budget and expenses of the Association for the first nine months of 1953 showed that operating expenses will be very close to the budget.

Mr. Teas then reported on the finances of the 1953 Annual Meeting. There was a deficit of $533.57 over receipts. Council approved that this be paid from general funds.

The Convention Committee has a balance of $1,412.46 in their treasury as of November 10, 1953. Council by an unanimous vote approved that this money be held by the committee to be used for future meetings.

The Secretary reported that thirty-four contracts for Journal advertisements in 1954 had been received; there are twenty-six still to be heard from. It is anticipated that the situation in 1954 will be equal to 1953.

The committee which was organized to cooperate with Leather Industry of America was discharged. The President is to write to the chairman and express appreciation of their efforts.

The Editor reported on the papers he has.

Council has aked that the Editor prepare proper forms to—

1. acknowledge all papers
2. an order form for reprints.

The President asked Council to consider the desirability of the Association underwriting the expenses of a delegate to the International Union Meeting, if there is no one going otherwise. The matter was laid on the table for future consideration.

It was announced that Dr. Turley has been elected First Vice-President of the International Union of Leather Chemists' Societies, and this means that he will become President in 1957.

The Nominating Committee has submitted the following slate of candidates.

President-Elect:-

Mr. E. B. Thorstensen — Mr. G. W. Schultz

Council:-

Mr. Dominic Meo — Dr. W. C. Henry
Mr. M. Maeser — Mr. W. R. Lotz

Mr. Paul D. Jost was elected to active membership.

Council approved the acceptance of the invitation from the Textile Chemists Association to participate in the Perkins Centennial meeting in 1956.

Mr. D. Meo, chairman of the Convention Committee, discussed the plans for the 1954 Meeting at Bedford Springs and Council approved the plans.

Council approved the selection of the New Ocean House, Swampscott, Massachusetts for the 1955 Annual Meeting for dates late in May.

The President has appointed the following members to serve on the Awards Committee: Mr. M. Maeser as chairman, Messrs. D. Lord and J. Ivany.

The matter of additional activities other that the Annual Meeting was discussed and it was generally conceded that the various Production Clubs serve such purpose.

Some thought might be given to a small meeting in Winter where only research papers are to be presented. The matter was taken under advisement.

The next meeting of Council will be held at the Sylvania Hotel in Philadelphia on February 11, 1954.

The meeting was adjourned at 3:30 P. M.

Respectfully submitted,

FRED O'FLAHERTY, *Secretary.*

ALSOP AWARD COMMITTEE

The President of the Association announces the appointment of the following members as constituting the Alsop Award Committee.

M. MAESER, *Chairman*
DANIEL LORD
JOHN IVANY

International Union of Leather Chemist Societies

REPORT FROM THE BARCELONA CONFERENCE

THE PHYSICAL MERITS OF LEATHER

The Conference wishes to draw attention to the following inherent properties of leather which have been studied by the members of its constituent Societies and which make leather a unique and valuable material. The Conference does not wish to imply that all leathers have all these properties to the same degree. Part of the art of leathermaking is to accentuate those properties needed to enable the leather to fulfill the particular purpose for which it is intended.

High tensile strength, exemplified in the use of leather for transmission, belting, and safety belts.

Resistance to tear. This is derived from the three-dimensional weave of the fibers. It is this three-dimensional weave which is of value in shoe making because it provides stitch tear resistance.

High resistance to flexual fatigue. This is a property which makes leather suitable for belting, and is of great value in ensuring that leather gives long service in gloves and footwear.

High resistance to puncture. This is also derived from the three-dimensional weave of fibers and is shown to a high degree by sole leather.

Good frictional properties under wet and dry conditions. These are imporant for leather belting which will grip a pulley but will slip in an emergency. Leather soles play a part in lessening fatigue when walking because they grip the pavement and road surfaces.

Controlled area stability. Sole leather and many other leathers are made dimensionally stable. Gloving leather, however, can be so made that it will stretch.

Low bulk density. Because of its fibrous nature, the bulk density of leather is low without impairing other properties.

Good heat insulation. As its low bulk density indicates, there is a considerable amount of air in the interstices between the fibres of leather. This air clings to the fibre surfaces, and such stationary air is a poor conductor of heat, an imporant factor in bodily comfort.

Permeability to water vapour. So far as is known, leather fibres will hold more water vapour when exposed to air at any given relative humidity than any other fibers, whether natural or synthetic. This property makes leather a valuable covering for the foot, since leather can absorb perspiration either as liquid or in vapour form. The perspiration absorbed will pass through the leather and be dissipated in the outside atmosphere.

Thermostatic properties. Like all other hygroscopic materials, leather resists temperature changes. When it is heated part of the heat absorbed is utilized to volatilize water vapour. When cooled, some of the heat lost is replaced by the heat liberated when the cooled leather absorbs water vapour. Because the water vapour content of the air is constant, or changes only slowly, the atmospheric temperature determines when the leather absorbs water vapour. When the temperature is falling, the relative humidity of the air is increasing: the leather breathes in water vapour, and heat is liberated which counteracts the fall in air temperature. Conversely, when the air temperature is rising, the relative humidity is decreasing and the leather breathes out water vapour which causes it to lose heat, and hence counteracts the rise in air temperature. When leather "breathes", therefore, it counter-

acts temperature changes: to the foot or body a delayed temperature change contributes to comfort.

Large internal surface area. As previously stated, the large internal surface area of leather contributes to its heat insulating properties. It also contributes to its permeability to water vapour, since it ensures that the leather reaches equilibrium with the moisture in the air between the fibers very quickly. It is of special importance in leather clothing and footwear because it assits the replacement of the moisture which leather loses to the atmosphere.

Mouldability. Leather is plastic and can be moulded and will retain its new shape. This property is invaluable in hydraulic leathers and in the manufacture of footwear. A leather shoe becomes moulded to the shape of the foot.

Resistance to water. When required, leather can be made water resistant without losing its permeability to water vapour.

Resistance to fire. Leather resists heat and flame, and for this reason is used for protective clothing.

Resistance to fungi. Leather can be made resistant to mildew by the use of very small amounts of various fungicides.

Barcelona, September, 1953.

REPORT OF MEETING OF TECHNICAL COMMITTEE CHAIRMEN

A meeting of the Technical Committee Chairmen was held Sunday, June 7, 1953, at the Netherland Plaza Hotel for the purpose of reaching a common agreement with respect to the format of the methods of the Association and the general coding of such methods.

After considerable debate it was the concensus that the methods should employ the same manner and paragraph numbering system as typifies the Federal Specification KK-L-311a.

A system of coding, for the Methods Booklet, involving the use of a suitable letter of the alphabet to associate with a given series of analyses, was described. Some of the chairmen expressed approval of such a system, others indicated preference for the present lettering system, while still others believed the Federal Specifications method of coding should be followed. It was agreed that whatever system was approved by the Editorial Committee would be suitable to the Technical Committee Chairmen.

The meeting was concluded with a request by the Technical Committee Coordinator that each chairman submit a report covering the activities of his Committee through June 1953.

Respectfully submitted,

E. S. Flinn,
Coordinator of Technical Committees

REPORT OF THE VEGETABLE LEATHERS COMMITTEES

We are pleased to submit herewith a general report of the 1952-53 activities of the ALCA Hide Powder Committee and the Tannin Analysis Committee. This report will be amplified and more details given in the reports submitted by Mr. Arthur N. Kay and Mr. I. D. Clarke, the Chairmen of these Committees.

The Hide Powder Committee

The Hide Powder Committee completed examination of a new lot of hide powder prepared by Mr. Frank Marshall and has found it satisfactory. Mr. Marshall has been authorized to distribute this powder as ALCA Official American Standard Hide Powder, Lot No. 26. (See April 1953 issue of Journal.)

The Tannin Analysis Committee

Mr. Kay has supplied the following preliminary report and advises that he will send his completed report on the tannin analysis methods as soon as he has approval of his Committee members.

"*Report on Activities of Tannin Analysis Committee* 1952 - 1953

"During 1952, the principal activity of this committee, both under the chairmanships of Mr. Stanbery and the undersigned, has been the revision of the methods. This has been carried out by a sub-committee but, at one time or another, most of the members of the full committee have been consulted. The revised methods are now in the hands of this sub-committee for final checking and approval. Since time is pressing, it will not be possible to obtain the approval of the full committee before submitting the revision to Council, though copies will be sent to all as soon as the sub-committee passes thereon.

"It had been hoped to have made a study of the effect of drying time on the results of the analysis, but that has not yet been possible. This study is planned for the coming year.

"There has been some divergence of opinion, between this committee and the Federal Specifications Board, as to the number of units to be sampled from a given shipment, and a start was made to try to find a sound basis for the Sampling Table. Both for lack of time and of information, as well as Council's expressed desire to put all Sampling into a separate section, that study is far from complete. Subject to Council's advice as to whether such a study comes under our jurisdiction, under the circumstances, it is hoped to continue with this during the coming year."

If I have been correctly informed, further work on the determination of nitrogen in leather has been requested of the National Bureau of Standards. I have received no report on these studies.

The question of a review of the ALCA methods of vegetable leather analysis has been raised. No request, however, has been received from the Council for such a review and rewriting of these methods to bring them into harmony with the other ALCA methods that are now being rewritten.

Your Chairman feels that the Chairmen, Mr. Clarke and Mr. Kay, and their respective committees are to be congratulated for having done excellent work. Mr. Kay and his Committee are to be specially commended for completing the heavy task of revising and rewriting the ALCA Methods for Tannin Analysis.

Respecfully submitted,

JEROME S. ROGERS, *Chairman*
Vegetable Leathers Committees

REPORT OF THE MINERAL LEATHERS COMMITTEE

In 1953 the Mineral Leathers Committee undertook two projects, an investigation of the so called overall basicity [1], and a study of methods for the determination of zirconium in leather.

The undersigned, as the chairman of the committee, reported on the conclusion of the first phase of this work at the committee meeting held at the Netherland Plaza in Cincinnati on June 6, 1953.

The term overall basicity, which was coined by the late Professor E. Theis of Lehigh University, denotes the total acid combined with the chrome complex either in a tan liquor [2] or in chrome tanned leather [1]. In most cases the overall basicity of leather should be considerably lower than the sulfate basicity as obtained by the ammonia or phosphate methods [3] because the former would include not only other anions besides SO_4 but also syntans and dyestuffs. In leathers which are low in these last named substances the figures for both basicities naturally would be much closer together.

The committee members received duplicate samples of an undyed ground chrome leather on which to determine the neutral and total SO_3, as well as the overall basicity; the latter includes a chrome determination for which the provisional ALCA method recommends several versions of the wet oxidation method [4]. It was left to the individual analyst which of these he would follow.

In addition the members were asked to determine the overall basicity of dyed chrome leather. Here too a Cr determination was, of course, necessary for the purpose of computing the basicity. The results can only be considered preliminary because they constituted for most participants the first attempt to use the new method. It is felt that a publication of individual returns by the eight committee members who cooperated in this study would not serve any useful purpose. So much may be said, however.

1. The intra-laboratory agreement is in all instances superior to the agreement between the laboratories.

2. The agreement between laboratories regarding chrome content is surprisingly poor. It may well be that inaccurate standardization is the principal reason of these disparities. The committee decided at the convention to enclose a weighed amount of standard sodium bichromate with the next samples and to give more explicit directions for the determination of their chrome content.

3. The inter-laboratory agreement concerning sulfate basicity suffers from the widely divergent figures for chrome. In some instances the differences are accentuated by the results for combined sulfate which depend on accurate figures for neutral as well as total sulfates. The former fluctuate much more widely than the latter, possibly because of their very minuteness (comprising a few mg. only).

4. The inter-laboratory agreement for overall basicity is many times poorer than that for sulfate basicity. One analyst would conclude from his figures that the leather he examined would have no more than traces of any anions besides SO_4, whereas others would attribute to such anions nearly 50 per cent of the total acid groups.

The committee reached the conclusion that the main difficulty arises from the fact that owing to the absorptive capacity of the leather digest at pH 7.5 a steady slow stream of standard alkali can be added without upsetting the equilibrium. Evidently more stringent conditions must be worked out before the method can be successfully tested in inter-laboratory work. R. Stubbings and the chairman volunteered to study this problem and to submit their findings to the committee later on.

Table of Results

Obtained by Zirconium Sub-Committee on Colored and White Leather

	Somerville Wendkos	Strymish	Axelrad	Seligsberger
Sample No. 1				
Cr_2O_3%	5.6	5.54	5.10	5.51
	5.6	5.55	5.36	5.42
ZrO_2%	4.7	4.96	4.90	5.17
	4.7	4.97	5.07	4.83
Sample No. 2				
ZrO_2%	16.0	20.7	16.59	14.94
	16.2	20.8	16.53	16.02
TiO_2%	0.7	0.8	1.12	1.06
	0.7	0.8	1.16	1.15

A sub-committee of five members studied the method of Somerville and Wendkos [5] for the determination of zirconium in leather. The findings which they reported are summarized in the table below. The results are in fair agreement when moderate amounts of chrome and zirconium occur in leather. However, in the second sample where large amounts of Zr are present together with some TiO_2, analysts reported difficulties in dissolving the fused ash and the figures obtained by some of them are at considerable variance with the rest.

At the meeting R. Stubbings suggested utilizing in a new series of tests the wet oxidation method for the digestion of Zr leather and estimating Zr while keeping the Ti in solution as pertitanate. In the method of Somerville and Wendkos Ti must be determined colorimetrically in an aliquot of the solution obtained from the fused ash. In á second portion Zr and Ti are precipitated and ignited together.

Bibliography

1. L. Stubbings and E. R. Theis, *J.A.L.C.A.* **48,** 155 (1953)
2. Thorstensen T. C. and E. R. Theis, *J.A.L.C.A.* **47,** 588 (1952)
3. Report of Mineral Leather Comm., *J.A.L.C.A.* **47,** 452 (1952)
4. Methods of Sampling and Analysis of the ALCA, page D2
5. Somerville and Wendkos, *J.A.L.C.A.* **48,** (1953)

Respectfully submitted,

L. Seligsberger, *Chairman*
Mineral Leathers Committee

REPORT OF PRE-TANNING AND POST-TANNING OPERATIONS COMMITTEE

Recommendation for revision of methods of analysis of (a) beamhouse liquors, and (b) fats, oils and related materials, were submitted to the Council at its mid-winter meeting, 1952. These recommendations included deletion of some existing methods, minor changes in others, and the adoption of several additional methods for analysis of fats, oils, etc., taken from the methods of the A.O.C.S.

It was pointed out that it would be necessary for an editorial committee to prepare a complete text of the revised methods, if and when these changes, additions and deletions were approved, since it is impossible for each committee to do this if it is desired that our methods be uniform with respect to style and arrangement, unless the committee chairmen are given precise instructions as to the form desired, which has not been done.

It is my understanding that these reports were approved by the Council a year ago, but I have no information as to what steps, if any, have been taken towards completing the editorial revision, and submitting such of the changes as require the approval of the membership to the vote of the members.

There still remains the task of revising the various miscellaneous methods, pertaining to the analysis of acids, alkalies, salts, etc. I hope to complete this task before the next annual meeting.

Respectfully submitted,

H. B. MERRILL, *Chairman*
Pre-Tanning and Post-Tanning Operations Committee.

REPORT OF TANNERY PRACTICES COMMITTEE

A. Engineering Practices – R. K. Blakely, *Chairman*
No current activity.

B. Industrial Waste Disposal Committee – J. F. Wagoner, *Chairman.*
No current activity.

Respectfully submitted,

REUBEN G. HENRICH, *Chairman*
Tannery Practices Committee

REPORT OF THE STANDARDS AND SPECIFICATIONS COMMITTEE

The Standards and Specifications Committee has been quite active during 1952 and 1953.

During the past two years this Committee has dealt almost exclusively with Federal Specification KK-L-311, Leather and Leather Products, Methods of Sampling and Test. Methods covering physical tests in these specifications have, by agreement, been assigned to Mr. M. Maeser as Chairman of the Physical Testing Committee. Only methods and sampling for chemical analysis have been considered by this Committee.

The following sections of Specification KK-L-311 have been under consideration:

Section 6,000; *Chemical Analysis of Leather*:

The Committee recommendations and changes in the original version of this section were again presented in detail at a meeting of the Federal Specifications Board in Washington, D. C., on March 6, 1952. Some of the proposed changes differing from the methods of the ALCA have been investigated by Committee work before approval. There is no major change in this section not approved by this Committee. The full details of all recommendations to the Federal Specifications Board concerning

Section 6,000 are set forth in a letter to the Committee Members dated April 12, 1952. This specification has been published.

Section 7,000; *Tannin Analysis*:

The original proposal by the Federal Specifications Board concerning this section differed in many details from the method of the ALCA. This Committee worked in collaboration with the ALCA Committee on Tannin Analysis, under the chairmanships of G. W. Stanbery and A. N. Kay, in order to bring all proposals up to date and to include several changes proposed by the Tannin Analysis Committee. The final recommendations were sent to the Federal Specifications Board under date of July 23, 1952, with joint approval of this Committee and the Tannin Analysis Committee. Since there are a few points still open in the ALCA revision, Mr. Everett Wallace, of the Federal Specifications Board, recommended that this section be temporarily deleted from KK-L-311 in order to expedite its promulgation.

Section 60; *Methods for Sampling, Inspection and Test*:

This section was discussed in detail prior to and also during the meeting of this Committee in Swampscott, Mass., on June 1, 1952. The committee did not approve this section on the grounds that the data on which it was based did not include sole leather. This was the only instance during the year when the Standards and Specifications Committee did not agree with the Federal Specifications Board on a major point. Mr. Everett Wallace, of the Federal Specifications Board, recommended that "an exception be made in the commodity specification for sole leather requiring the use of the present three sampling positions for chemical analysis until such time as we may have more supporting data to warrant a change". (July 15, 1952)

It is to be noted that the action of this Committee refers only to sole leather where Section 60 is applicable to sole leather. No other branch of the leather industry had voiced any objection to this section or brought any matter pertaining to it to the attention of this Committee.

Similar proposals covering the change in sampling position for bends from the three positions to one single position were received by the Committee through Tanners' Council dated August 1, 1951. Considerable opposition developed to this proposal and it was finally withdrawn early in 1952. A study of the sampling locations for sole leather was proposed by the Bureau of Standards, Leather Section, in order to settle this question. No further report has been received regarding the disposition of this matter.

Proposals were received by this Committee recommending minor changes in the hide substance requirements of Federal Specification KK-L-261-c,

Sole Leather. This matter was discussed in Committee and approved, and recommendations forwarded to Tanners' Council in a letter dated June 1, 1952, for official action.

Proposed specifications for insoles have been under consideration by the Committee, covering both straight vegetable tanned insoles, and alum and chrome retanned insoles. A proposal to add formaldehyde retanned insole leather was tabled until more data on this subject are available.

R. Lollar has presented a study to the Committee on the precision of methods for the analysis of leather. The Committee considered this work during its meeting on June 1, 1952, and recommended to the Council of ALCA that this matter be studied in detail by an appropriately assigned committee.

During 1953, up to the date of the annual convention, the committee has considered proposed changes in the Specification for Leather, Sole, Vegetable Tanned, KK-L-261-c. A recommendation has been made to change the degree of tannage requirements in order to compensate for the change in analysis brought about by the use of chloroform instead of petrolic ether. Some changes were also made for purpose of clarification. It was also recommended that the scrap obtained when cutting soles should be accepted as a specimen for chemical analysis, if the cutting is done in the presence of a Government Inspector.

Proposed amendment to Harness Leather Specification, KK-L-171-d, presented to the Chairman, did not contain matters concerning chemical analysis or requirements, and is therefore outside the scope and activity of this committee.

One member has resigned and one new member has been added to the committee. The present membership is as follows:

R. G. Ashcraft	Kenneth H. Knight
G. Arthur Brown	R. M. Lollar
Sverre Dahl	Charles W. Mann
Arthur N. Kay	Elwood E. Presley

Respectfully submitted,

Carl G. Telander, *Chairman*
Standards and Specifications Committee

REPORT OF THE LEATHER FINISHES COMMITTEE

The Leather Finishes Committee activity in 1952-1953 has centered on the development of methods as follows.

1. Determination of flexibility and adhesion of our finish on upper leather for shoes.
2. Measurement of tack on finish of leather.

3. Detection of nitrocellulose in finish on leather.
4. Determination of total solids in leather finish.
5. Determination of ash in leather finish.

Even though tentatively approved by the Committee, the methods are being edited for wording for final presentation.

Other methods for testing leather finishes are being considered by the Committee. However, progress has been slow principally because regular business has not permitted sufficient time.

Respectfully submitted,

C. P. McKee, *Chairman*
Leather Finishes Committee

REPORT OF THE MECHANICAL LEATHERS COMMITTEE

The first and organizational meeting of the Mechanical Leathers Committee of the ALCA was held at the Bismarck Hotel in Chicago on Friday, February 20, 1953. Those present were:

Alfred S. Berens—Chicago Rawhide Manufacturing Co.
Donald C. Bundy—Northwestern Leather Co.
Robert W. Hague—J. E. Rhoads & Sons
Howard L. Hartman—C. W. Marsh Co.
Arthur Hirsch—Albert Trostel & Sons Co.
Henri Krakowski—Chicago Belting Co.
John C. Schneller—Chicago Rawhide Manufacturing Co.
Jack H. Van Sciver—Shingle Leather Co.

The meeting was called to order at 9:30 A. M. with Mr. Berens presiding. In view of the chairman's agreeing to write and publish the minutes of the meeting, no secretary was appointed.

The members introduced themselves and gave a brief description of the work done by their respective companies and by themselves, individually, in their various capacities.

A brief description of the background of the Mechanical Leathers Committee was given by the chairman. It was agreed by the Committee that in view of the natural domination of the ALCA by the shoe and allied trades, a Mechanical Leathers Committee has long been needed. The hope was expressed that the present committee would form the nucleus of ever increasing activity of the Association in the field of mechanical leather products.

The pertinent sections of the ALCA bylaws and, in particular, Article X on Technical Committees, were read so that the committee members would be aware of the Association regulations governing committee activity.

The purpose of this organizational meeting was discussed and seemed to involve answering four basic questions.

1. Should an ALCA committee on Mechanical Leathers be established in fact?
2. What would be the scope of such a committee's activities?
3. What would be the extent of member participation?
4. How can committee activities best be correlated with individual company self interest?

It was agreed that the answer to question (2) was most important and if adequately answered and agreed upon would almost automatically answer the other three questions. Therefore, the question of the scope of the Mechanical Leathers activities was discussed in considerable detail. It was agreed by the committee that four major fields of activity seemed to be of most immediate concern. These were:

A. The Improvement of Mechanical Leather

A number of characteristics of mechanical leather which could (and should) be improved beyond the presently available standard of quality were listed as follows:

1. Heat resistance
2. Corrosion (of metals)
3. Tensile strength
4. Elongation
5. Tear resistance
6. Abrasion resistance
7. Compression set
8. Porosity
9. Dimensional Stability
10. Flexibility
11. Moldability
12. Ease of cutting
13. Bursting strength

The committee was in unanimous agreement that the improvement of the heat resistance of leather was the most important single problem facing the mechanical leather industry. It was generally (but not unanimously) agreed that the corrosion of metals by leather was second in importance, but the other properties were not listed in order of their importance.

B. Classification of Mechanical Leather Specifications

It was agreed that this Committee should also function in an effort to classify and reduce the number of specifications covering mechanical leather. This would include not only those originating from Government agencies but also those originating from individual civilian customers.

C. Improvement of Impregnations for Mechanical Leather

It was agreed that the Committee's contacting the chemical industry with this problem on a Committee basis (rather than on an individual com-

pany basis) might stimulate more activity in those companies to produce materials suitable for our use. It was also agreed that the Committee could seek out impregnations not now generally used, since our knowledge of presently available chemicals is frequently determined by what the chemical industry chooses to advertise or by what their sales representatives think we need.

Certain basic properties of mechanical leather impregnations were outlined as follows (again not necessarily in order of their importance):

1. Oil insolubility
2. Heat stability
3. Flexibility (at least to -70° F.)
4. Sharp melting point or low impregnation viscosity
5. Non-corrosive
6. Ease of handling
7. Moldability
8. Ease of cutting
9. Non-toxic

D. Standardization of Physical and Chemical Tests for Mechanical Leathers

This would necessitate the development and evaluation of standard methods for examining almost all the properties listed as desirable improvements of leather. Again, tests on heat resistance were considered to be the most immediate in importance, with suitable tests in both water and a variety of oils being considered vital to mechanical leather progress. Various specific test procedures were also briefly considered; it was apparent that a number of laboratories were proceeding independently to work on problems which, if properly coordinated, would be of great value to the industry as a whole.

In order to proceed immediately with these agreed upon functions of the Mechanical Leathers Committee, three subcommittees were appointed.

SUBCOMMITTEE I—Mechanical Leather

(This subcommittee will be responsible for all Mechanical Leathers Committee activities in the field of improvement of leather as well as the classification and reduction of existing specifications.)

Arthur Hirsch, *Chairman*
D. C. Bundy
J. C. Schneller
Arnold Horween—Horween Leather Co.

SUBCOMMITTEE II—Mechanical Leather Impregnations

(This subcommittee was to be responsible for all the Committee activities in the field of leather impregnation,)

A. S. Berens, *Chairman*
H. L. Hartman
Henri Krakowski

SUBCOMMITTEE III—Physical and Chemical Testing

(This group was to be responsible for the development and standardization of physical and chemical tests.)

R. W. Hague, *Chairman*
J. H. Van Sciver
J. T. Eaton—E. F. Houghton & Co.
W. D. Johnson—Graton & Knight Co.

After having outlined these major functions of the Mechanical Leathers Committee and having agreed upon three subcommittees to pursue these activities, there seemed to be no question as to the need for a Mechanical Leathers Committee or for a specific discussion of whether the Committee should in fact be established. It seemed obvious that member participation, at least based on the reactions of those present at this meeting, could be counted on in view of the many problems facing the mechanical leather field.

It was felt that the Mechanical Leathers Committee should be somewhat larger than was the case at this initial meeting, with representatives of several more packing manufacturers and tanners being included. Allied trade representatives should also be invited to participate in special meetings.

The latter part of the meeting was devoted to the discussion of specific problems, primarily in the field of physical testing. General agreement was reached on a number of specific tests although they are to be investigated thoroughly by Mr. Hague's committee. For example, it was agreed that all boil tests and shrink temperature tests were to be run in distilled water of known purity. These boil tests were to be run for two hours at 100°C with the leathers being placed in actively boiling water, and the shrinkage determined parallel with the backbone after 24 hours conditioning at room temperature. It was agreed that the moisture content of mechanical leather at the time of test was of extreme importance and that all physical and chemical tests should be run on the basis of a standard temperature and per cent relative humidity preconditioning.

It was agreed that whenever cooperative tests are run by committee members as many of the following analytical items as possible would be reported:

1. Source of hide
2. Type of hide
3. Type of tannage
4. Cr_2O_3
5. Ash
6. Hide substance
7. Degree of retannage
8. Grease content
9. Stuffing or fat liquoring (or both)

10. Preconditioning (temperature and percent relative humidity)
11. Moisture
12. Chlorides
13. Sulfates
14. Buffed and unbuffed
15. Method and rate of drying (if known)
16. Location of test samples

It was agreed that the Mechanical Leathers Committee would hold its next meeting in Cincinnati in conjunction with the ALCA's Fiftieth Anniversary Convention in June of 1953.

The Chairman was directed to express the Committee's apprciation to the Chicago Rawhide Manufacturing Co. for their financial support of this initial meeting.

The meeting adjourned at 4:00 P. M.

Respectfully submitted,

ALFRED S. BERENS, *Chairman*
Mechanical Leathers Committee

FIRE RESISTANCE OF LEATHER

DEFINITION:

Fire resistance is defined as resistance to combustion, resistance to support of combustion after the ignition flame is removed, and resistance to afterglow due to progressive exorthermic char oxidation.

1. *Scope*

The method is intended for use on light leathers, and especially for upholstery leather. The Vertical Bunsen burner test is especially valuable where measurement of after-glow is of primary interest. The 45° Microburner test is generally applicable where both after-flaming and after-glow are significant.

2. *Specimens*:

The specimens shall be:

2.1—A rectangular piece of leather 2 x 12.5 inch, for the Vertical Bunsen burner test.

The orientation of the specimens relative to the backbone shall be noted.

2.2—A circular piece of leather 6 inches in diameter, for the 45° Microburner test, punched with five 3/16" inch holes to fit the holder studs.

3. *Apparatus*:

The apparatus shall consist of:

3.1—A Columbia Flame Test Cabinet, (Fig. 1), or its equivalent, described by Little, who has also discussed the problem of flame resistance

(Robert W. Little, Editor, "Flame-proofing Textile Fabrics", Reinhold Publishing Corp., New York, 1947, page 111 ffd.).

FIRE RESISTANCE OF LEATHER

SPECIMEN HOLDER

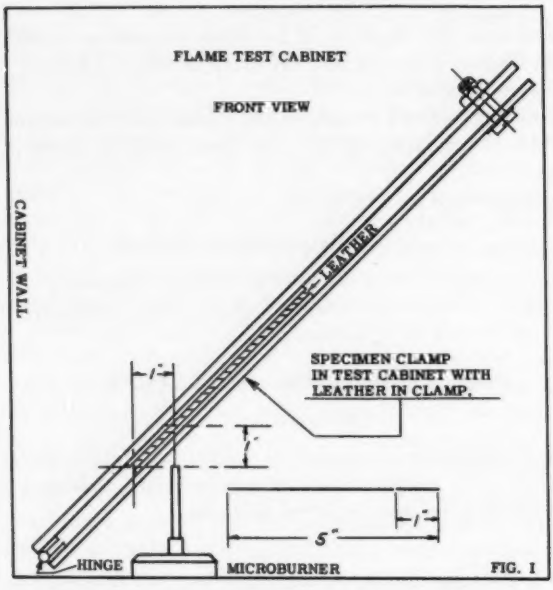

FIG. I

SPECIMEN CLAMP - OPEN

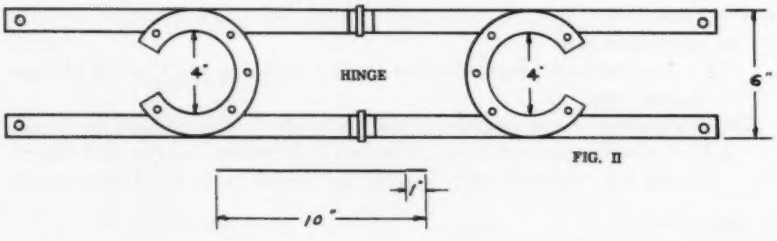

FIG. II

The inside dimensions of the wooden cabinet shall be 12 inches wide, 12 inches deep, and approximately 25 inches high. The front of the cabinet is

completely closed by means of two vertical-sliding pyrex glass doors. Ventilation is afforded by means of nine 0.5 inch holes along the bottom of each side and the 6 inch hooded vent in the top. The microburner is securely fastened to a square wooden block which is set in a fixed position in the cabinet. Notched metal strips, screwed to the sides of the cabinet, serve to keep the specimen racks in constant alignment with the burner. The gas for the microburner is held at a nearly constant pressure by means of a reservoir. A water manometer shall be used to indicate the relative gas pressure. When the manometer is placed (inclined) at an angle of 15° to the horizontal, a difference reading of approximately 22 mm. shall be maintained. A small pilot light is permanently attached to the microburner. Moving the heat source once the specimen is in position for test should be avoided.

3.2—Specimen Holder

A 6 inch circular sample holder (Fig. II) is used in the 45° Microburner test, otherwise the Columbia test cabinet is used without modification.

The circular specimen holder is made from two ⅛ inch annular brass rings, 6 inch external diameter, 4 inch internal diameter. They are converted to a horseshoe shape by removing a sector whose internal circumferential length is 1.5 inches. These rings are mounted on a rack to hold the sample in the proper position in the test cabinet. One of the rings carries short ⅛ inch diameter carriage bolts placed around the circumference while the other ring carries similarly aligned holes. Thus the two rings can be clamped over the leather specimen. This clamp holds the leather in a planar form throughout the test.

3.3—A dead weight type of thickness gage as specified in the method for thickness.

3.4—A triple beam laboratory balance graduated to read to 0.01 grams.

3.5—A stop watch.

3.6—A Bunsen burner (Tirril type) 0.375 inch inside diameter.

4. *Procedure*:

4.1—Vertical Bunsen Burner Test

The test specimen shall be weighed to 0.01 gram and the thickness measured to the nearest 0.001 inch at the end of the specimen nearer to the tail. The specimen shall be suspended from a clamp in the top of the test cabinet with the long dimension vertical and the tail end down. The lower end of the specimen shall be attached by two springs to the bottom of the cabinet to hold it 0.75 inch above the top of a Bunsen burner. The luminous flame, 1.5 inches long, secured from gas burned with the air ports of the burner closed, shall be applied for 12 seconds. The after-flaming and after-glow shall be the time from the end of the test flame application period until the after-flaming and after-glow have ceased. Finally, the piece shall be removed and the vertical char length and weight loss determined.

4.2—45° Microburner Test

The circular test specimen shall be weighed to the nearest 0.01 gram, and mounted in the horseshoe shaped clamp so that the finished side will be against the flame during the test, and so that the diameter parallel to the backbone passes through the center of the open end of the horseshoe clamp. The assembled clamp shall be inserted in the test cabinet so that the leather is at a 45° angle to the base of the cabinet and so that the inner edge of the clamp is 0.75 inch above the top of the microburner. The position of the sample clamp is also controlled so that the center of the burner is 1 inch in from the outer edge of the clamp. This positioning of the burner and clamp will cause the 1.75 inch flame to strike the leather 1 inch from the burner, and at a spot on the leather about 0.4 inch from the edge of the clamp (see Fig. I) with the flame playing up the slope of the leather. The flame in the burner is slightly tipped with yellow and fuelled by butane, giving a temperature at the point of contact of about 800°C. The horseshoe shaped clamp shall be placed so that the open side is at the end diametrically opposite from the point of flame application, thus leaving about 4.5 inches (6 inch sample, less 1 inch clamp less about 0.5 inch between clamp and area of flame contact) for flame travel. The flame shall be applied to the leather for twelve seconds, a stop watch being started at the ignition of the test flame. The time before a hole appears in the leather, and the length of after-flaming and after-glow measured at the conclusion of these two periods shall be recorded. Then the specimen shall be removed from the holder and re-weighed to establish the weight loss (due to grease and moisture loss as well as combustion), and the vertical length of char shall be measured.

5. *Results*:

The results of the vertical Bunsen burner test shall record the leather thickness in mils, the weight loss in grams, the char length in inches, and the after-flaming and after-glow in seconds.

The results of the 45° microburner test shall record the leather thickness in mils, the weight loss in grams, the vertical char length in inches, the time for a hole to appear in seconds, and the after-flaming and after-glow in seconds.

6. *History and Status*:

This method was developed by the Fire Resistance Sub-committee of the A. L. C. A. Physical Testing Committee; published in 1950, J. Amer. Leather Chemist's Association, **45** 715 (1950). See also Flammability of Clothing Textiles, ASTM, part 7, page 186. The Tester may be obtained from U. S. Testing Company.

Received, October 23, 1953.

A Flex Tension Test for Leather*

By T. J. Carter and J. R. Kanagy

Leather Section
National Bureau of Standards

Abstract

A flexing machine operating under tension has been developed for the testing of leather. The machine may be used to detect differences in flex lives of leathers caused by tannage, lubrication, accelerated aging, and deterioration.

Studies were made on the influence of axial load, bending angle, diameter of roller and effect of temperature on the flex life of leather. The flex life was found to be practically a straight-line relation of the axial load and the log of the endurance in flexes. Duration of time of flexing decreased as the angle of bend increased. The logarithm of the number of flexes before break is a straight-line function of the diameter of the roller in inches, and a plot of the log of the flexing endurance against the reciprocal of the absolute temperature gives an approximately straight-line relation. The heat of activation is related to the energy of flow and indicates that the mechanism of degradation is based to some extent on flow which increases with increase in temperature.

1. Introduction

The tendency for leather to crack in a shoe vamp is well known. This "crackiness" is generally caused by flexural fatigue. According to J. H. Dillon [1] the term fatigue in the broadest sense denotes change in a property of a material or structure taking place in a finite interval of time under a given set of conditions." Conditions, of course, include temperature, relative humidity, and chemical environment, which includes gaseous content of the air surrounding. Fatigue may be further defined as that phenomenon wherein a material ruptures or changes one or more of its properties permanently after a measured "fatigue time" during which a system of finite stresses has been imposed.

Fatigue phenomena are essentially time phenomena, and in the case of dynamic fatigue, heat is developed by the hysteresis mechanism which raises the temperature above that of the surroundings. Furthermore, seldom is a fatigue test conducted in the absence of reactive agents such as oxygen. Hence, careful classification and definition of state are particularly necessary in discussing fatigue phenomena.

It is desirable to consider briefly the various physico-chemical phenomena that can occur during a controlled fatigue operation, namely, in a sample of a fibrous material held for a period of time under stress in air at known but possibly varying temperature. The major effects may be listed as (a) molecular flow or secondary bond slippage resulting only in change of shape, (b) physical rupture of chains by stress concentration in a localized region, (c) orientation of chains with or without crystallization, (d) crystallization by

*This report is made as a part of the Leather Research Program sponsored by the Research and Development Branch, Military Planning Division, Office of the Quartermaster General, Department of the Army. This program is under the Advisory Direction of the National Research Council.

cooling or orientation, (e) second order transitions, (f) chemical scission of chains, and (g) cross linkage. Leather, held under stress, changes in shape; therefore, there must be molecular flow and secondary bond slippage. It stretches; therefore, there must be some orientation of chains. There also is cracking of the grain, followed on increasing the stress by complete failure of the fibers; therefore, there must be physical rupture of chains by stress concentration.

Fibers, in general, are more resistant to fatigue than rubber and plastics. Some results given by J. H. Dillon [1] for single filaments bent through 180° cyclically to fracture under one gram tension are given below:

	Average Number of bends
Wool and nylon	20,000
Cotton	3,200
Natural silk	1,800
Soybean fiber	150
Cellulose acetate	100
Viscose	75
Casein fiber	23
Glass fiber	1

A single leather fiber would be expected, from similarity in chemical composition and crystallinity, to approach the resistance to fatigue of wool or nylon. However, for a natural material, such as a piece of leather, where the fibers are woven at random into a fabric-like material, the effect of internal friction on fatigue would be highly important. The internal friction would depend upon tannage, lubrication, and fiber structure. Fatigue of leather also depends upon previous history, especially treatment prior to tanning, and environment during use. An important example of use environment is the cracking of a shoe vamp which undoubtedly is accelerated by adsorption of perspiration.

The excellent flexing endurance of good leather is well known. This property of leather is superior to that of most other materials and is one of the reasons leather is so highly desirable for use in shoe uppers. A test for the measurement of this property in leather is, however, difficult to develop because of the long duration of time necessary to reach an end point.

An important study of the resistance of leather to breakdown during flexing was made by M. Maeser [2]. He pointed out that as leather is flexed it becomes less stiff and also that it is less notch sensitive than metals and plastic. To overcome the fact that the leather becomes less stiff on flexing, he developed a machine which tested a specimen through a cycle in which the flexing span was progressively increased. The maximum span did not, however, stretch the leather beyond its point of permanent set; even with notched

specimens, the only end point obtained in the test was the cracking of the grain.

It has been shown by the Institute of Paper Chemistry [3, 4] that the bending endurance of paper is critically decreased by the application of tension to the specimens under test. The application of tension is necessary to keep a specimen of a material having a high permanent set under constant stress. The adaptation of a procedure having this desirable characteristic to leather appeared to have possibilities. This paper discribes a flex-tension machine operated under a practically constant axial load and also a discussion of some of the results obtained with it on the evaluation of upper leather.

2. Apparatus and Procedure

A sketch of the type of flexing mechanism developed for these tests on leather is shown in figures 1 and 2.

The two rollers x and y are mounted horizontally on ball bearings about ¾ inch apart in a flexing head which reciprocates. The ball bearings allow the rollers to rotate freely over the leather and prevent the development of excessive heat by friction during the flexing of the leather. The roller assembly which fits into the flexing head may be rotated so that the angle at which the leather is bent may be varied. In sketch A the leather is bent at an angle

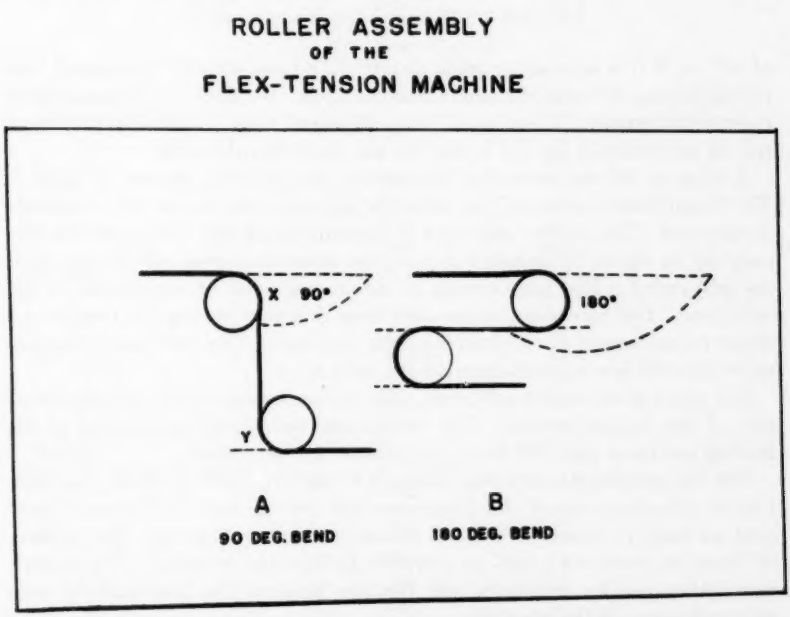

FIGURE 1.—Diagram of the roller assembly of the flexing machine.

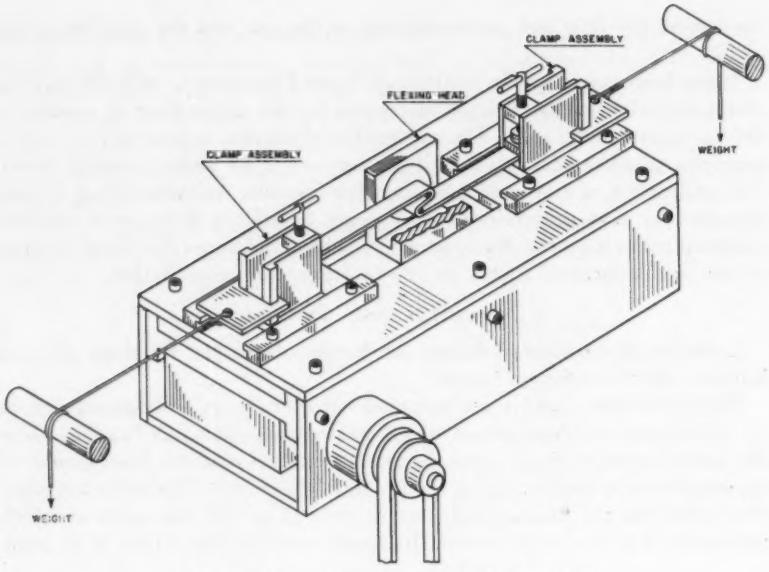

FIGURE 2.—Diagram of the flexing machine.

of 90°, in B it is bent at an angle of 180°. The assembly is so designed that rollers having different diameters may be used. Weights are placed at both ends of the leather. These axial loads, of course, must be equal so that there will be no tendency for the leather to slip towards either side.

A diagram of the assembled flex-tension apparatus is shown in figure 2. The flexing head consists of the reciprocating unit in which the roller assembly is mounted. The leather specimen is passed around the rollers and the two ends (A in figure 1) clamped in the jaw assemblies from which the loads are suspended. The jaws are set at point zero prior to application of the axial load. The movement of the jaws from this point during the test gives a direct measurement of the stretch of the specimen. The maximum displacement for each jaw is approximately 1⅝ inches.

The speed of the motor is 60 rpm. The reciprocating motion is in the direction of the applied tension. The rollers pass over about two inches of the leather specimen and 120 flexes per minute are produced.

The flex-tension machine was designed to test only one specimen at a time. Unless otherwise stated the conditions for testing were a 15-pound axial load, an angle of bend of 180°, and rollers $^{3}/_{16}$ inch in diameter. The number of flexes to grain crack and to complete failure was recorded. The stretch was determined by measuring the distance between the jaws and the zero reference point on the machine.

TABLE I

Effect of Load on Flexing Endurance of Degreased Retanned Upper

Axial load	No. of flexes before Grain crack	No. of flexes before Break	Elongation	Thickness
lb.			%	mil
	2400	8880	29	
	2200	9240	26	
6	1500	6960	23	
	1020	9960	26	
	900	8280	26	
	1140	5160	32	
Average	1530	8080	27	90
	300	4320	29	
	300	4080	26	
9	240	3660	29	
	180	2520	32	
	120	3900	29	
	240	3660	29	
Average	228	3700	29	90
	72	1440	34	
	68	3180	29	
12	88	2880	32	
	102	1980	34	
	35	2160	32	
	30	3360	29	
Average	66	2500	32	89
	23	600	40	
	4	1980	43	
15	10	2340	40	
	12	2340	43	
	3	1260	43	
	3	960	46	
Average	9	1580	43	88
	45	840	34	
	50	1620	29	
18	9	1320	34	
	6	840	40	
	50	1680	34	
	4	1800	34	
Average	27	1350	34	89
	7	660	46	
	3	1200	34	
21	4	1080	37	
	1	1080	34	
	3	1140	40	
	2	840	33	
Average	3	1000	37	90

The leather used in the tests consisted of the experimental upper leather prepared under sponsorship of the Office of the Quartermaster General. Specimens ½ by 8 inches were cut from the sides close to location 13 (see [3]). The average thicknesses of the specimens ranged from 5 to 6 ounces.

3. Results and Discussion

Studies were made on the effect of axial load and direction of cutting the specimen, effect of degreasing the original samples, the influence of lubricants, and the effect of accelerated aging on the resistance to breakdown during flexing.

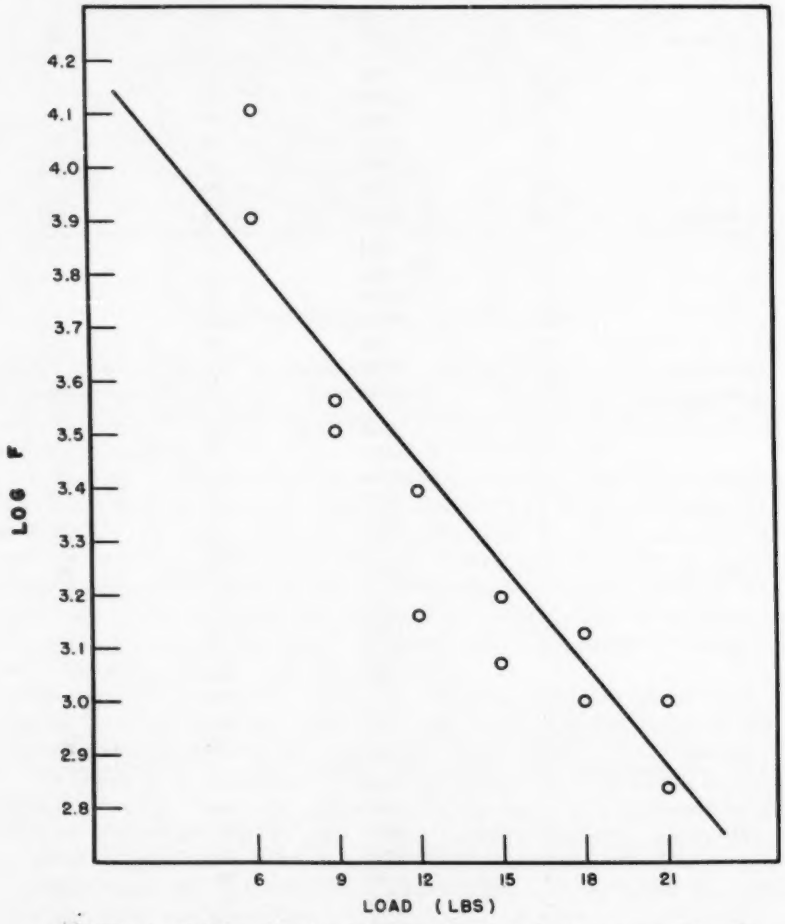

FIGURE 3.—Relation of load and the logarithm of the flexing endurance (Log F).

3.1 *Influence of Load*

The effect of axial load on the flexing endurance of greased and degreased chrome-retanned leathers was determined with 6–, 9–, 12–, 15–, 18–, and 21–pound axial loads. The results for the degreased leathers are given in table 1 and the results for both greased and degreased leathers in figure 3. It was found that the log of the number of flexes was related roughly linearly to the axial load. These results are in agreement with findings when similar tests were applied to paper [3], [4]. It was decided to use a 15-pound axial load in all

TABLE 2

Effect of Angle of Bend on the Flexing Endurance of Retanned Upper Leather

Bending angle in degrees	Number of flexes				Elongation, %		Thickness
	At grain crack		At break		At break		
	Greased	Degreased	Greased	Degreased	Greased	Degreased	
							mil
45	1140	300	17520	6240	32	20	87
	1080	270	16200	5640	32	17	94
	480	720	16800	6120	32	20	89
	4800	390	30240	7560	32	23	89
Average	1875	420	20190	6390	32	20	90
90	67	49	6360	2100	37	23	88
	120	80	6540	2760	34	23	90
	780	75	9000	1800	32	21	93
	53	90	8220	2340	40	23	93
	70	95	6840	1740	32	26	88
	33	366	5400	2220	40	26	88
	540	89	6120	2400	34	29	87
Average	238	121	6930	2200	36	24	90
135	180	7	8400	1380	40	34	91
	84	10	7080	1380	49	32	88
	28	49	6960	1980	40	29	89
	180	12	8940	1320	40	32	88
	90	12	6660	1560	43	34	90
	35	28	6180	1860	40	26	92
	210	8	5400	1560	40	32	90
Average	115	18	7090	1580	41	31	90
180	20	5	8400	2040	37	29	91
	10	1	7080	1440	40	40	92
	5	5	5760	1920	43	29	92
	5	2	5460	1080	40	34	90
	2	3	6240	1440	43	37	92
	40	2	8400	1080	46	32	87
	7	4	6480	1320	43	34	92
Average	13	3	6830	1470	42	34	91

subsequent tests. The fifteen-pound axial load was about the midway point of the series of different loads studied and it did not too greatly prolong the time for obtaining an endpoint.

3.2 *Effect of Bending Angle*

The angle of bend over the rollers may be varied as described above by rotating the roller assembly in the flexing head. Measurements of the influence of bending angle on the flexing endurance were made at 45°, 90°, 135°, and 180°. The results for greased and degreased leather specimens are given in table 2.

For an angle of 45°, the number of flexes at grain crack and at break are highest. The number of flexes at break for greased leather at angles of 90°, 135°, and 180°, are equal. For degreased leathers there is no change in flexing endurance above 90°. Elongation at break for both greased and degreased leathers continuously increased with an increase in the angle.

The variation of thickness in the specimens might appear to have appreciable influence on the flexing endurance of leather since the elongation of the

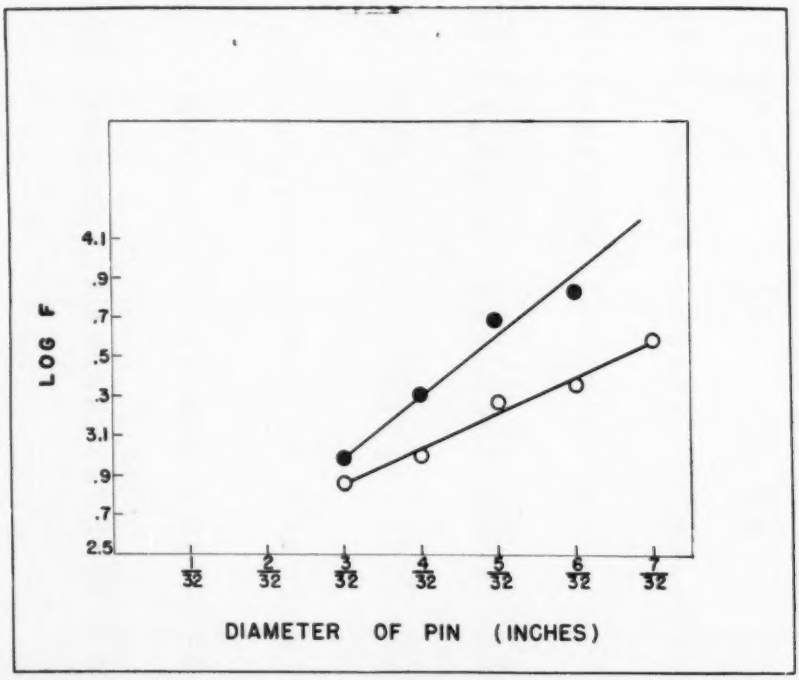

FIGURE 4.—Relation of the diameter of pin and the flexing endurance (Log F). ● leather containing grease, ○ leather containing no grease.

specimens varies with the thickness and the increased extreme fiber bending stress. However, experimental results show that thickness is not an important factor in flex life.

3.3 *Diameter of Roller*

The size of rollers studied ranged from $^{3}/_{32}$ to $^{7}/_{32}$ inch in diameter. The results are given in figure 4 and tables 3 and 3a. The plot of the logarithm

TABLE 3

Effect of Diameter of Roller on Flexing Endurance of Degreased Upper Leather

Diameter	Number of flexes before		Elongation at break	Thickness
	Grain crack	Break		
inches			%	mil
3/32	1	600	34	81
	1	720	26	80
	1	720	37	82
	1	840	32	84
	1	720	37	82
	—	—	—	—
Average	1	720	33	81
4/32	1	720	32	80
	1	1200	37	80
	1	1560	34	80
	1	600	29	81
	1	1440	26	79
	—	—	—	—
Average	1	1100	31	80
5/32	1	2220	29	85
	1	2580	32	85
	1	1440	29	80
	1	1320	32	79
	1	1800	32	79
	—	—	—	—
Average	1	1870	30	81
6/32	1	2760	34	79
	1	2400	34	81
	1	2040	34	76
	1	2040	32	79
	1	2280	29	78
	—	—	—	—
Average	1	2300	32	79
7/32	1	3480	29	81
	1	2760	29	81
	1	4920	29	79
	1	5340	29	80
	1	2700	32	81
	—	—	—	—
Average	1	3840	30	80

TABLE 3A

Effect of Diameter of Roller on Flexing Endurance of Upper Leather Containing Grease

Diameter	Number of flexes before Grain crack	Break	Elongation at break	Thickness
inches			%	mil
3/32	2	960	40	87
	4	840	46	85
	65	840	40	80
	7	1200	37	95
Average	19	960	41	87
4/32	2	1080	40	83
	40	2160	46	80
	150	2040	43	80
	24	2880	37	90
Average	54	2040	41	83
5/32	430	5040	37	83
	3	4920	43	90
	33	5760	34	85
	150	4560	43	82
Average	150	5070	39	85
6/32	140	5040	42	82
	50	9360	29	92
	10	6360	40	91
	100	5760	40	81
Average	75	6630	38	87

of the number of flexes to break against the diameter of the roller in inches gives a linear relationship.

Elongation at break changes only slightly with a change in the diameter of the roller. The number of flexes to grain crack showed no significant variation when the diameter of the roller was increased.

3.4 *Influence of Temperature on the Flex Life of Leather*

The effect of temperature on the flexing endurance of typical leathers was determined at 11°C, 22.2°C, and at 34.0°C. These results were plotted in figure 5 against the reciprocal of the absolute temperature; from these plots the heats of activation shown in table 4 were calculated. The plot of the log of the flexing endurance against the reciprocal of the absolute temperature gives a straight line relation. The heat of activation is related to the energy of flow and indicates that the mechanism of degradation is based to some extent on molecular flow which increases with an increase in temperature. Molecular flow or secondary bond slippage was listed above as one of the

effects when a sample of a fibrous material was held under stress for a period of time. The rather low heat of activation, which averaged only about 2.5 K calories, indicates that molecular flow for leather may not be a major effect of fatigue. Flow, of course, is of much greater significance for the nonfibrous materials.

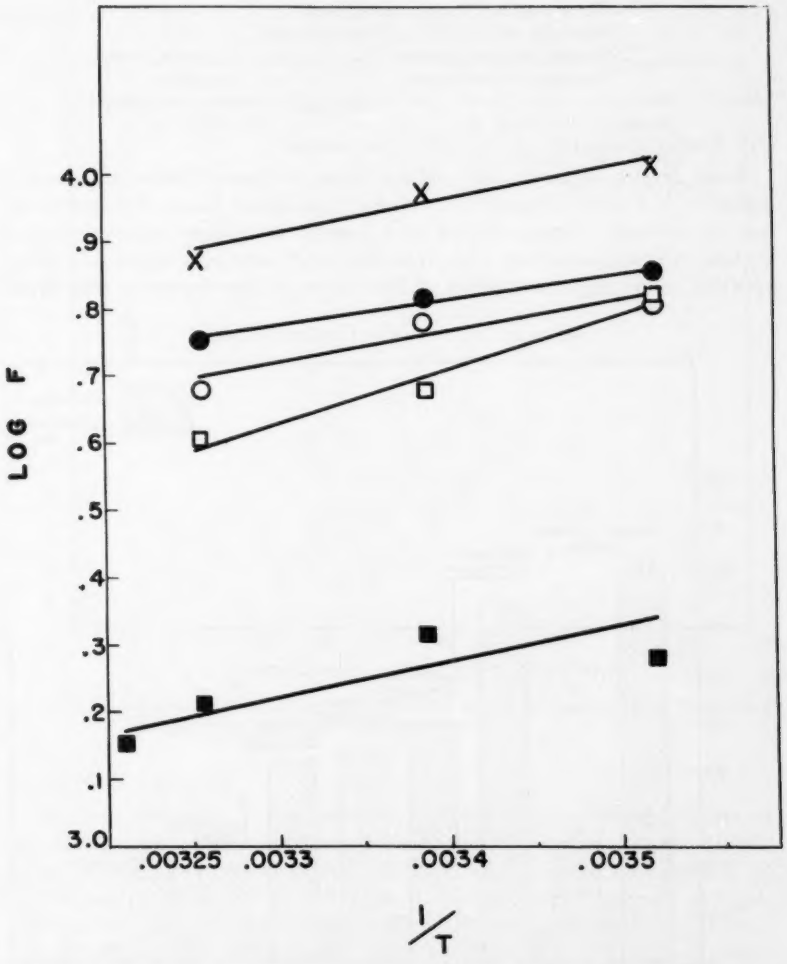

FIGURE 5.—Effect of temperature on resistance to breakdown on flexing. × chrome tan, ● chrome retan, ○ chrome tan, □ chrome retan, ■ vegetable tanned.

TABLE 4

Heats of Activation for Flexing Fatigue of Leather

Sample	Heat of activation
	K calories
Chrome-tanned leather (fatliquored)	2.4
Chrome-tanned leather (fatliquored)	2.6
Vegetable leather retanned with chrome	1.8
Chrome-retanned leather	3.8
Vegetable-tanned leather	2.2

3.5 *Flexing Endurance of Typical Upper Leathers*

Some results obtained with various types of upper leather are given in figure 6. It is shown that the resistance to breakdown during flexing is greatest for straight chrome-tanned and lowest for straight vegetable-tanned leather. Chrome-retanned with vegetable and various syntans are intermediate. The important effect of lubrication is also shown in this figure.

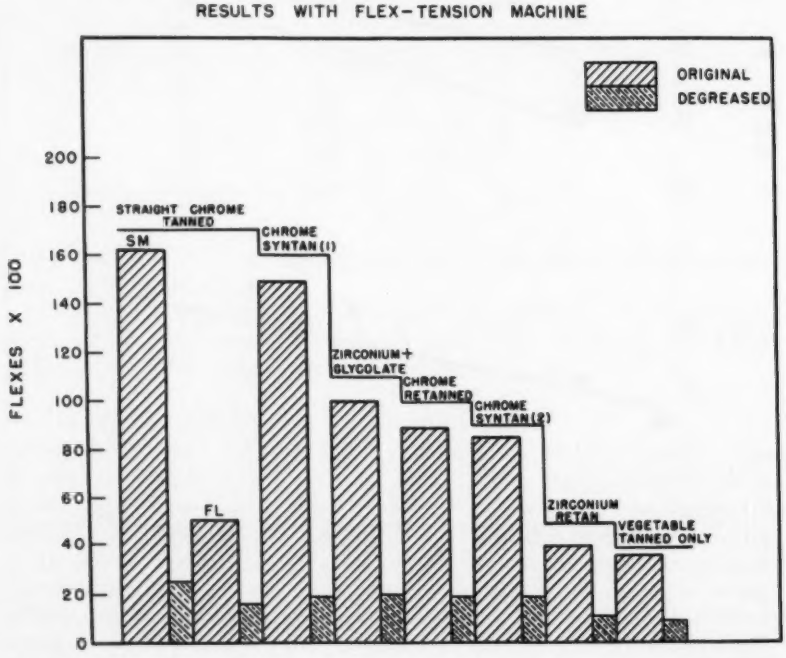

FIGURE 6.—Results obtained with various types of leathers on the flexing machine. SM Stuffing mixture, FL Fatliquored.

For all types of tannages the resistance to breakdown is greatly decreased by degreasing the specimens. Fatliquored chrome leather shows less flexing endurance than stuffed chrome leather.

As shown by the results given in figures 7 and 8, the resistance to breakdown on flexing is inversely porportional to the percentage of vegetable tannins. This is indicated with leathers containing grease and with degreased leathers.

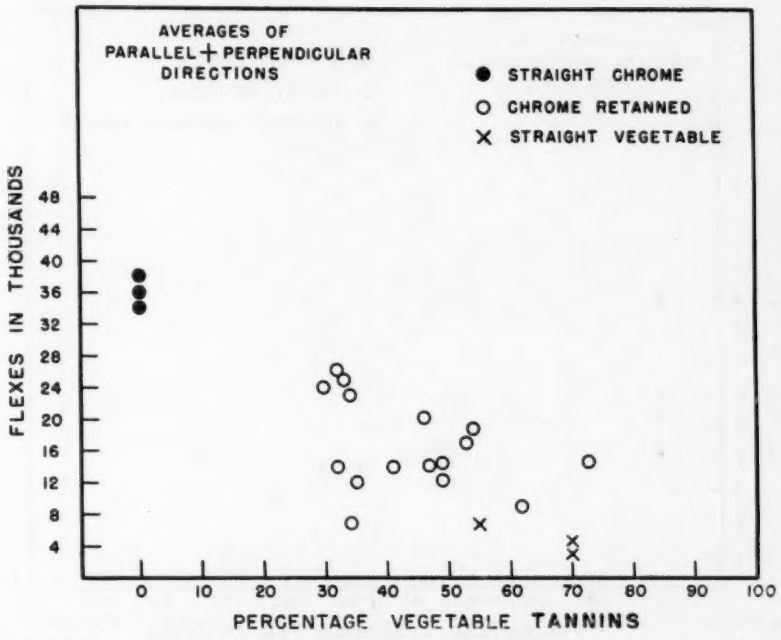

FIGURE 7.—Influence of percentage of vegetable tannin on resistance to breakdown during flexing for leather containing grease.

3.6 *Effect of Lubrication*

The large difference in the resistance to breakdown on flexing of greased and degreased leathers suggests that lubrication is a factor of prime importance. For this same reason, degreased leather was retreated with mineral oil, cod oil, and regular stuffing mixtures, each dissolved in chloroform to give different concentrations. The concentrations were so controlled that from 2 to 69 per cent of the lubricants were added based on the original weight of the samples. The results are given in figure 9. Each point represents the average of six measurements.

Stuffing mixture was found to be superior to cod or mineral oil in improving the resistance to breakdown during flexing. This is probably due to the greater viscosity of the stuffing mixture which consequently is less readily removed from the leather by the flexing action. For all three kinds of lubricants, the resistance of the leather to breakdown was increased to a maximum at a definite percentage content of the lubricant after which there was no further increase.

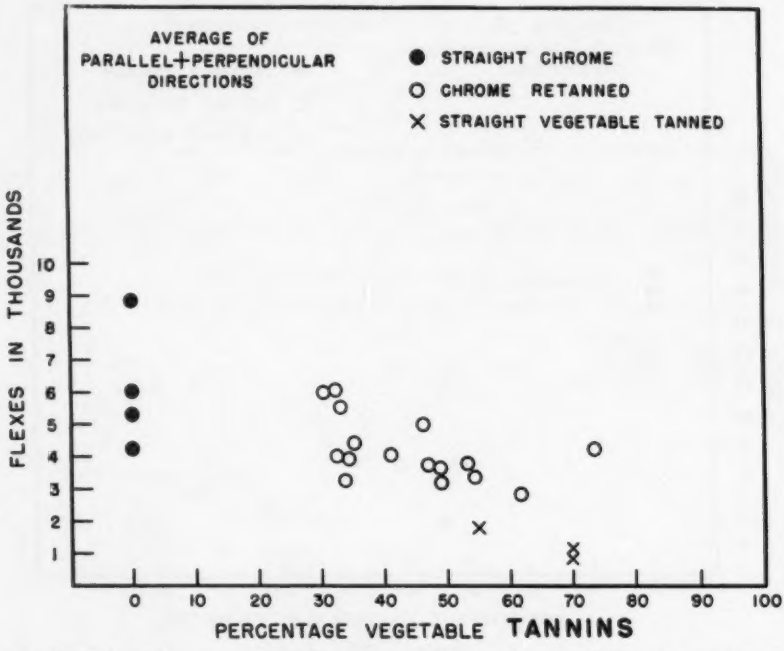

FIGURE 8.—Influence of percentage of vegetable tannin on resistance to breakdown during flexing for leathers degreased.

3.7 *Effect of Accelerated Aging*

Studies were made on the sensitivity of the flex-tension machine as a criterion for the measurement of the resistance of leathers to accelerated aging. These results should also give an indication as to its sensitivity to the measurement of deterioration. The results shown in table 5 for leathers aged at 100°C indicate that flexing endurance decreases progressively with time of aging. Each group of samples was cut from approximately the same location of different tanned sides. Data are given for specimens cut in both the parallel and the perpendicular direction. Specimens cut in the parallel direction are

more highly resistant to breakdown on flexing. Degreased specimens show lower percentage decreases in resistance than greased specimens; however, it must be remembered that the latter have much higher values initially. The grease does not appear to offer any protection under accelerated aging conditions.

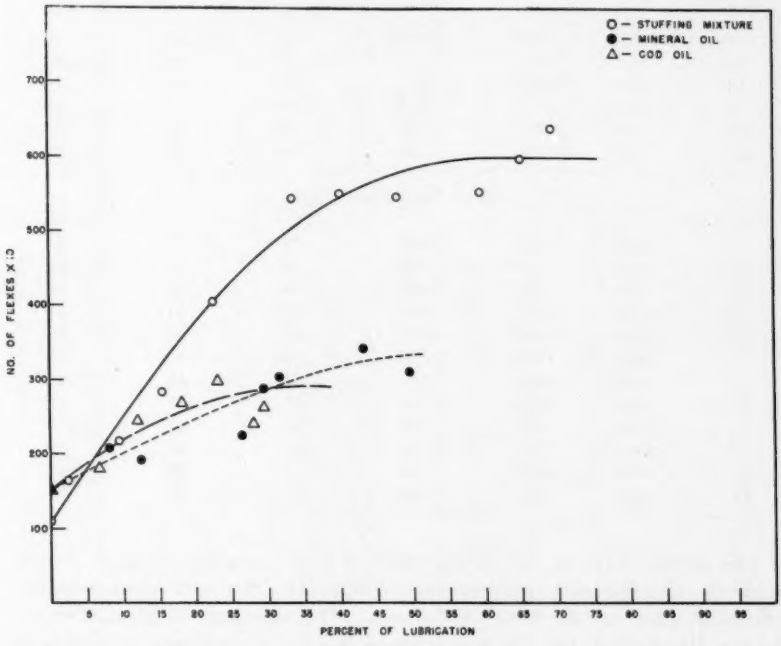

FIGURE 9.—Effect of percentage of grease on resistance to breakdown of leathers on flexing.

3.8 *Effect of Water*

The effect of water on flexing endurance was studied with chrome, chrome-retanned, and vegetable-tanned leathers. The specimens were wetted by soaking them in distilled water for a period of time ranging from 6 to 24 hours. The results showed that the variations of the soak periods produced no differences in the flex resistance. The total water adsorption for chrome-tanned leather containing grease was 45 per cent, and that for fatliquored chrome-tanned leather was 72 per cent based on the conditioned weight of the specimens. The moisture content of the specimens during flexing was maintained by applying water to the surface at a rate of one drop per minute.

TABLE 5

Effect of Accelerated Aging on Reistance of Leather to Breakdown During Flexing
Chrome Retanned Leather

Aging period	Flexes before break			Elongation, %		
	With grease		Degreased	With grease		Degreased
hr	//	⊥	//	//	⊥	//
0	13110	9480	2490	37	53	28
24	6740	4500	2630	37	46	30
48	5560	4580	1930	40	64	34
72	2670	2450	1480	38	54	29
120	2550	2810	1540	38	46	30
168	1130	730	1240	38	60	31
	Leathers Tanned with Syntans					
0	14470	4050	1990	54	77	33
24	1780	140	840	53	57	34
72	660	150	460	50	60	34
0	11350	5270	2650	45	57	33
24	3140	1100	2270	45	40	26
72	2310	1480	720	43	50	31
0	9350	5860	2590	41	42	32
24	7860	3650	1370	35	37	35
72	5380	3290	750	36	42	33
0	6910	5950	1550	45	48	35
24	6780	5690	670	42	49	37
72	2360	2340	370	52	49	32

The results of the studies on the effect of water are given in figure 10 and table 6. Chrome and vegetable-tanned leathers differ with respect to the effects of water on the flexing endurance. The resistance to breakdown by flexing for chrome and chrome-retanned leather is increased as shown in figure 10 by the treatment with water whereas that of the vegetable-tanned leather is decreased as shown in table 6.

4. General Discussion

In considering the mechanism of the action in the flex-tension machine, interest arose with regard to the amount of heat produced in the leather by flexing. It was postulated that sufficient heat might be produced to cause deterioration. Since vegetable-tanned leathers showed lower resistance to breakdown in flexing than chrome-tanned leathers, it was thought that a shrinkage phenomenon might be involved. Accordingly, the temperature of the leather specimen was determined in the region of flexing by a thermocouple imbedded in the leather. It was found that the maximum temperature reached for both lubricated and nonlubricated specimens was approximately

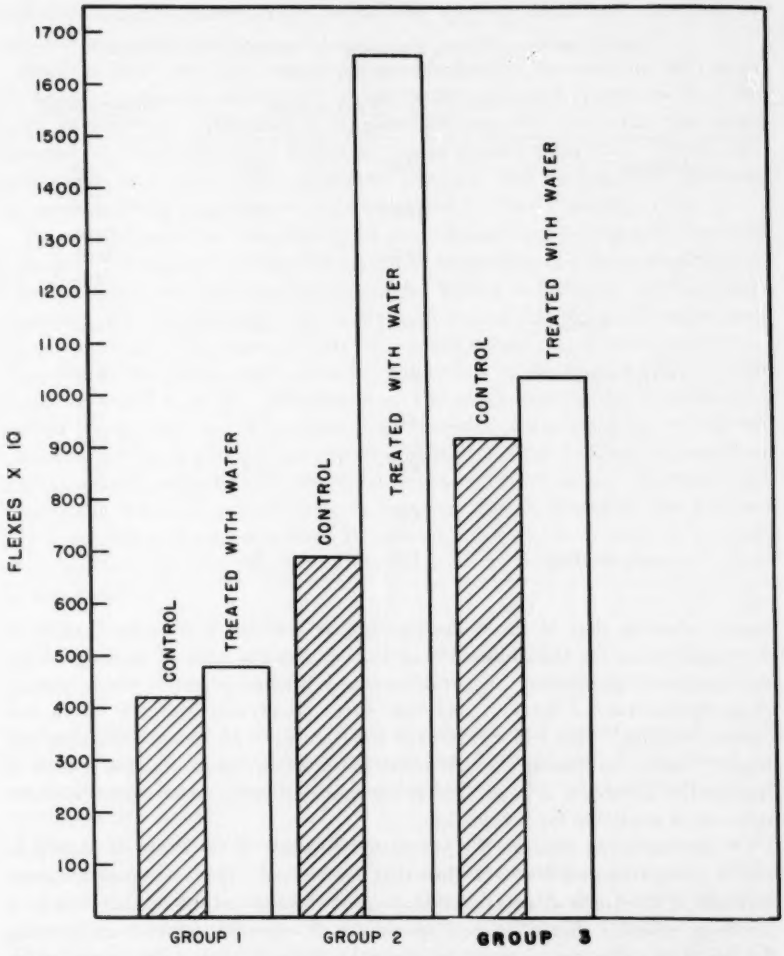

FIGURE 10.—Effect of water on resistance of leathers to breakdown during flexing. Group 1, fatliquored chrome; Group 2, chrome retanned with Exan; Group 3, chrome retanned and stuffed.

40°C. This temperature is considerably below the shrinkage temperature of both vegetable- and chrome-tanned leathers.

The influence of water on the flexing endurance of chrome and vegetable-tanned leather differs. The resistance to breakdown during flexing for chrome and chrome-retanned leather is increased, figure 10, by the treatment with

TABLE 6

Effect of Water on Flexing Endurance of Vegetable-tanned Leather
Vegetable-tanned leather

	Flexes to break		Flexes to break
	With grease	Degreased	With grease
Original	1440	360	2760
			2760
	1800	240	3360
			4440
	1440	—	2040
			3480
Average	1560	300	3140
Treated with water	600	30	840
			720
	840	136	720
			900
	840	90	900
			540
	600	—	840
Average	720	85	760

water, whereas that of the vegetable-tanned leathers is decreased, table 6. An explanation for this difference in behavior is the ease of wetting of the two types of specimens. Vegetable-tanned leather is much easier wetted than chrome-tanned leather, and the water penetrates into the fibers and causes swelling. This will tighten the fibers to such an extent that they are readily broken on flexing. On the other hand, since chrome-tanned leather is less readily wetted, it is possible that the moisture remains between the fibers where it is available for lubrication.

Of interest is the data on the percentage increase in thickness of vegetable and of chrome-tanned leathers when they are wetted. The average percentage increase in thickness of several specimens of chrome-tanned leather was 15.1 per cent, whereas that of several specimens of vegetable-tanned leather was 7.7 per cent. The reason that the vegetable-tanned leather did not increase in thickness as much as the chrome-tanned leather was that the former had already been plumped to a greater extent because of the presence of the vegetable tanning materials.

The flexing machine as designed has three variables, namely, axial load, diameter of roller, and bending angle, which influence the rate of breakdown on flexing. The results show that the logarithm of the flexing endurance shows approximately linear relations with both the axial load and the diameter of the roller. The flexing endurance decreases with increase in axial load and

with decrease in diameter of the roller. The angle of bend has little effect at angles greater than 90°.

Angle of bend and diameter of roller undoubtedly have similar effects on the leather specimens under test. In the results obtained it appears that the angle of bend was influential at angles less than 90°, and that the roller diameter was the influential factor at angles greater than 90°. This is the reason that an angle of bend of greater than 90° had no apparent effect on the results. These results were obtained with a $^3/_{16}$ inch diameter roller.

There appears to be a similarity in the influence on flexing endurance of load and of diameter of roller since both of these factors give approximately linear relations with the logarithm of the flexing endurance. It has been pointed out by Schiefer and Boyland [6] with regard to work on textiles that the strain produced in fibers on bending is a function of the radius of curvature. Thus if R is the radius of bend and r_1 the radius of the fiber the fiber strain would be equal to r_1/R. Restriction of the movement of the fibers within leather would increse the extreme fiber stresses corresponding to a given curvature of the leather. The extreme fiber strain for bending a sample of leather where restriction of movement was complete would approach the value r_2/R where r_2 corresponds to one-half the thickness of the leather. For a specimen of upper leather 80 mils in thickness bent over a $^3/_{16}$-inch roller the fiber strain, considering complete restriction of movement, would be approximately:

$$\frac{r_2}{R + r_2} = \frac{0.04}{0.09 + 0.04} = \frac{0.04}{0.13} = 31 \text{ per cent}$$

If the strain exceeds the elastic limit of the fiber permanent set will occur. It may therefore be seen that the strain on the fibre may be increased by increasing the load or decreasing the diameter of the roller over which the specimens are flexed.

The mechanism of the breakdown in the flex-tension test appears to be based principally upon flow and on mechanical action. These are effects a and b, listed in the introduction of this paper, of the various physico-chemical phenomena which can occur during a controlled fatigue operation. The decrease in the flexing endurance with increase in temperature and the fact that an activation energy exists are evidence that flow is one of the causes of the breakdown on flexing. Most of the causes of failure must be based on mechanical action.

The effect of lubrication on the fibers is well known and needs no further explanation. The experiments with water where flex resistance was improved, figure 10, may be explained by the lubricating action of water. Water penetrates into the smallest capillaries and pores coating all of the finest fibrils with a layer of moisture which tends to decrease the friction between them.

Degradation in the flex-tension machine is probably based to a large extent on fiber fatigue caused by friction exerted by bending and axial stresses. The effect of tension is shown by the critical effect of increased axial load and decreased diameter of roller on the specimens. Vegetable tannins and other filling materials which go into the pores of the leather tend to prevent freedom of movement of the fibrils. Therefore, when a tension or bending stress is placed on the leather, the fibers are not free to overcome these stresses by rearrangement. Rearrangement would otherwise be permitted if more freedom of movement were allowed. For this reason, increasing concentrations of vegetable tanning materials increases breakdown on flexing and any other treatment which causes increasing tightness in the leather matrix will likewise increase the rate of breakdown on flexing.

5. Conclusion

A machine based on flexing under tension has been developed for the testing of leather. The machine may be used to detect differences in leathers caused by tannage, lubrication, accelerated aging, and deterioration. The test may possibly be used as a criterion for developing tannages and lubricants for leather as well as evaluating leather for use in shoe uppers and other uses where flexing is important.

Acknowledgment: The authors wish to express their appreciation to Walter K. Stone of the Textile Section for major contributions in the designing and construction of the flexing machine.

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Canaigre Investigations

X. FERMENTATION OF CANAIGRE LIQUORS BY YEASTS

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INTRODUCTION

In a previous paper [3] the need for removal of sugars to increase the purity of canaigre extract liquors was pointed out. A method for producing high purity tanning extracts was described in which the sugars are fermented by certain strains of *Aerobacter aerogenes*. This process has been carried out many time in pilot plant size lots of liquor (50 to 300 gal.) and has proved to be satisfactory for producing high purity extracts, except that no easily recoverable, marketable by-products are produced. According to theory [5] 50 per cent of the sugar fermented should appear as nongaseous products. The main non-gaseous product is 2,3–butanediol, which is found in fermented canaigre liquors in concentrations of up to 0.5 per cent [2] or about 65 per cent of theory. Several unsuccessful attempts make it seem very unlikely that this diol could be recovered economically during concentration of the liquors to powdered extracts. A further objection to the method is the formation of insolubles during fermentation [3], consisting of bacterial cells and some amorphous material, which require rather high speed centrifugation for removal.

Obviously it would be advantageous to find some organism that produces both a usable by-product and a high purity extract. To this end we are constantly searching for new types of microorganisms to ferment canaigre liquors. This paper deals with attempts to use yeasts for the fermentation.

MATERIALS AND METHODS

Yeast cultures which we have investigated may be divided roughly into 3 groups on the basis of their reactions to canaigre liquors:

1. Those that are killed or at least fail ro carry out any discernable metabolic process. This group comprises by far the largest number of the cultures tested.
2. Those that do not proliferate but do survive for a limited time and assimilate sugars.
3. Those that multiply and carry out normal metabolic processes.

Most of the work reported here pertains to members of the second group, which have been utilized according to a recently patented process [4]. Since yeasts are known to carry out fermentation processes without reproducing, it was reasoned that massive numbers of yeast cells might be used to remove

*One of the laboratories of the Bureau of Agriculture and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture.

sugars from canaigre liquors and thereby increase the liquor purity, The yeast would first be grown on a suitable medium, then the cells harvested and transferred to canaigre liquor long enough to assimilate the sugar. Recoverable ethanol would be produced in both stages. A search* was made for a yeast which would be comparatively insensitive to the toxic materials in canaigre. Some 60 strains were tested for their activity in increasing concentrations of a canaigre medium. Viability was determined by transfer to agar slants after each exposure. As the concentration of canaigre increased, most of the yeasts died. Eight cultures of group 2 were found which were capable of fermenting sugar in the presence of 6 per cent canaigre extract, equivalent to 3 per cent tannin. In addition, three species representing the third group have been isolated in this laboratory: two from canaigre and one from a sumac liquor. The last is a species of *Torulaspora*, which grows very slowly and destroys tannin and is therefore of no further interest. The first two will be discussed briefly at the end of the next section.

Canaigre liquors were prepared by water extraction of dried shredded canaigre roots. The spent residue remaining after extraction of the tannin always contains a large amount of starch. Since utilization of this material is of great importance in the canaigre development program, acid-hydrolyzed spent canaigre root was investigated as a possible source of nutrients for producing yeast cells. Hydrolysis was accomplished by boiling the ground spent residue under reflux in 2 per cent H_2SO_4 for some of the experiments. However, most of the material was prepared by countercurrent extraction with boiling 6 per cent H_2SO_4, followed by neutralization with calcium carbonate to pH 5–6 and filtration. The latter procedure was preferable for our purpose mainly because it yielded solutions with a higher sugar content. Absolute yield of sugar from the original starch, while of secondary importance in these studies, was close to theoretical.

Both liquors and hydrolyzates were sterilized in 100 to 500 ml. quantities, in flasks with capacities of about 5 times the volume of liquid to be used. All were incubated on a reciprocating shaker at 30° C. for the specified times unless otherwise indicated. Where cyclic fermentation was carried out, passing yeast cells from hydrolyzate to liquor and back again, equal volumes of each substrate were used. Inoculi were obtained by suspending in sterile water the growth from 24–48 hour agar cultures grown at 30° C.

Sugar in the liquors and hydrolyzates was determined by the method of Somogyi [6] after precipitation of the tannin and protein with lead acetate and the excess lead with sodium carbonate. Alcohol was determined on distillates with a dipping refractometer. Tannin analyses of the liquors were made by the official A. L. C. A. methods [1]. Yeast cells were estimated by plating or by direct microscopic counts.

*Dr. L. J. Wickerham, of the Northern Regional Research Laboratory, very kindly made this search among cultures in his collection.

TABLE I

Fermentation of Spent Canaigre Hydrolyzate* by Yeasts.
Incubated 18 hours on a reciprocating shaker at 30° C.

Yeast Culture	Sugar after fermentation %	Sugar fermented % of original	Alcohol gm/100 ml	Alcohol yield % Theoretical	
				Based on original sugar	Based on sugar consumed
NRRL Y 862 Saccharomyces cerevisiae	2.65	73.5	2.92	57.3	77.9
NRRL Y 821 " "	1.27	87.3	3.46	67.8	77.8
NRRL Y 791 " "	1.32	86.8	3.34	65.5	75.4
NRRL Y 813 " "	1.30	87.0	3.50	68.6	78.8
FYC " "	1.38	86.2	3.31	64.9	75.2
NRRL Y 359 Hansenula anomala	2.46	75.4	2.94	57.6	76.4
NRRL Y 660 Torulopsis utilis	2.57	74.3	2.86	56.1	75.5
NRRL Y 793 " "	2.66	73.4	2.82	55.3	75.4

*Contained 10.00% sugar; no added nutrient.

Results

The eight yeast cultures of group 2 were first tested for their ability to ferment the sugars and to produce alcohol in the hydrolyzates with and without supplemental nutrients. All the cultures grew well with some small differences in the rate of growth and the amount of alcohol produced. Table I shows the results of an experiment with unsupplemented hydrolyzate. After 18 hours' incubation 4 of the cultures had fermented about 87 per cent of the sugar and the other 4 about 74 per cent. Alcohol yields, based on the sugar consumed, were 75 to 79 per cent of theory.* An additional 7 hours permitted all the cultures to consume at least 84 per cent of the sugar, but the maximum was still about 87 per cent. Alcohol yields had not materially increased, and in some cases considerable quantities were lost.

Table II shows the results of fermentation of an hydrolyzate to which various nutrients had been added. The yeast used was Y 813. Potassium phosphate, magnesium sulfate and malt extract had no appreciable effect. Addition of ammonium phosphate or corn steep liquor resulted in the fermentation of slightly more of the sugar and the production of slightly more alcohol than in the unsupplemented control. However, the efficiency of conversion of sugar to alcohol was slightly lower with these two supplements.

*Calculated as 51 per cent of the wt. of sugar.

TABLE II

Fermentation of Spent Canaigre Hydrolyzate* with Added Nutrients by Yeast NRRL Y 813. Incubation 22 hours on a reciprocating shaker at 30° C.

No.	Nutrient Added**	Sugar after fermentation %	Sugar fermented % of original	Alcohol gm/100 ml.	Alcohol yield % Theoretical	
					Based on original sugar	Based on sugar consumed
1	None, inoculated control	2.42	77.9	3.87	69.2	88.8
2	$(NH_4)_2HPO_4$, 0.1%	1.66	84.9	4.01	71.7	84.6
3	K_2HPO_4, 0.05%	2.68	75.5	3.60	64.4	85.3
4	$Mg\ SO_4.7H_2O$, 0.01%	2.46	77.6	3.75	67.1	86.4
5	Corn steep liquor, 0.1%	1.71	84.4	3.95	70.7	83.7
6	Malt extract, 0.1%	2.57	76.6	3.69	66.0	86.2

* Contained 10.96% sugar.
** Wt./vol. basis.

Further experiments with other nutrient sources led to the tentative conclusion that supplemental nutrients are not necessary, except perhaps a small amount of nitrogen.

TABLE III

Cyclic Fermentation of Spent Canaigre Hydrolyzate* and Canaigre Leach Liquor by Yeast NRRL Y 660, *T. utilis*

Sample No. **	Incubation time hrs.	Sugar after fermentation %	Sugar fermented % of original	Alcohol gm/100 ml.	Alcohol yield % Theoretical		Yeast cells† millions/ml
					Based on original sugar	Based In sugar consumed	
H 1	24	0.47	83.0	0.92	65.2	78.6	133
L 1	none (control)	1.86	——	——			
L 2	6	0.82	55.9	0.48	50.5	90.6	109
H 2	24	0.43	84.5	0.95	67.4	79.8	156
L 3	16	0.06	96.8	0.77	81.1	83.7	96

*Contained 2.77% sugar.
**H=hydrolyzate; L=liquor.
†Plate count.

Since spent canaigre hydrolzates were found capable of supporting good growth of these yeasts with reasonable yields of alcohol, the next step was to harvest the cells, place them in a canaigre liquor and observe whether the sugars were feremented in the presence of tannin. Table III gives the results of an experiment in which a culture of *T. utilis* was used. It was first grown in an hydrolyzate for 24 hours. The fermented hydrolyzate was then centrifuged and the yeast cells transferred to a canaigre liquor. After 6 hours incubation, the fermented liquor was centrifuged and the yeast cells trans-

ferred to another sample of hydrolyzate. The process was repeated with another sample of liquor which was incubated for 16 hours, thus completing two cycles on each material.

In the hydrolyzates about 84 per cent of the sugar was fermented, and based on the sugar consumed 79 per cent of the theoretical amount of alcohol was produced. In the liquor incubated for only 6 hours, the fermentation was not complete. Only 56 per cent of the sugar had been consumed whereas in the liquor incubated for 16 hours, 97 per cent of the sugar was fermented. Alcohol yields from the liquors were 91 and 84 per cent of theory, respectively. Plate counts indicated that the yeast cells had increased in the hydrolyzates and decreased in the liquors.

TABLE IV

Tannin Analyses of Canaigre Leach Liquors
Fermented by Yeast NRRL Y 660, *T. utilis*

Sample	L 1	L 2	L 3
Incubation Time, hrs.	none	6	16
Soluble Solids %	5.41	4.45	3.61
Nontannin %	2.69	1.65	0.98
Tannin %	2.72	2.80	2.63
Purity 100T/SS	50.3	62.9	72.9
Total Sugars%	1.86	0.82	0.06

Table IV shows the tannin analyses of the fermented liquors. In the liquor fermented for 6 hours the purity (100 x Tannin/Soluble Solids) increased from 50 to 63 and in the 16 hour fermented liquor to 73. Since the analyses were run on centrifuged liquors there were no insolubles present. Tannin values did not change significantly.

An experiment in which an hydrolyzate containing 8.2 per cent sugar and a liquor containing only 0.8 per cent sugar were fermented by yeast NRRL Y 821 is reported in Table V. The hydrolyzate was inoculated with 3 million yeast cells per ml. After 19 hours' incubation there were 170 million cells per ml. by direct microscopic count and 41.5 per cent of the sugar had been consumed. After an additional 2 hours the cell count was 200 million/ml. and 57 per cent of the sugar had been fermented. At this point the cells were harvested by centrifugation and placed in the liquor, giving a cell count of 180 million per ml. After 5 hours 50 per cent of the sugar had been consumed; the cells were so badly clumped that they could not be counted. After an additional 20 hours, 75 per cent of the sugar had been consumed, the tannin content had not changed and the purity increased from 55 to 63. The harvested cells, incubated for 19 hours in a second aliquot of the hydrolyzate, increased to 450 million / ml. and consumed 79 per cent of the sugar. In a

TABLE V

Fermentation of Spent Canaigre Hydrolyzate* and Canaigre Liquor** by Yeast NRRL Y 821

Sample No.***	Incubation time hrs.	Sugar fermented % of original	Soluble solids %	Tannin %	Purity 100T/SS	Yeast cells† X 10^6
H 1	0	——				3
H 2	19	41.5				170
H 3	21	57.3				200
L 1	0	——	3.19	1.74	54.5	180
L 2	5	50.0				‡
L 4	25	75.0	2.63	1.65	62.7	‡
H 5	19	79.3				450
L 6	6	62.5				‡
L 7	22	62.5	2.55	1.61	63.1	‡

* Contained 8.2% sugar.
** Contained 0.8% sugar.
*** H=hydrolyzate; L=liquor.
† Direct microscopic count per ml.
‡ Could not count, cells clumped.

second aliquot of liquor, these cells consumed in 6 hours almost $^2/_3$ of the sugar, which amount was not increased by additional incubation. Alcohol yields in this experiment were of the same order of magnitude as those reported in the previous experiment.

Considerable attention has been given a new species of *Endomycopsis*,* a member of the third group of yeasts described in the preceding section. It has been isolated repeatedly from various canaigre preparations in which it grows very well. Unfortunately, it destroys tannin. Table VI shows a typical result when this yeast was used to ferment canaigre liquor. In this particular experiment there was a 12 per cent tannin loss. Many attempts, all unsuccessful, have been made to induce this organism to destroy the sugars in canaigre liquors without destroying tannin.

*Identified by Dr. L. J. Wickerham.

TABLE VI

Fermentation of Canaigre Liquor with *Endomycopsis sp.* 24 hrs. incubation with aeration at 30° C.

	Control	Fermented
Soluble Solids %	4.90	3.55
Insolubles %	0.24	1.08
Nontannin %	2.26	1.23
Tannin %	2.64	2.32
Purity Tan / SS X 100	53.9	65.4
Total Sugar %	1.1	0.06

TABLE VII

Fermentation of Canaigre Liquor with an Unidentified Yeast, Y 89
14 hrs. incubation with aeration at 30° C.

	Control	Fermented
Soluble Solids %	3.19	2.37
Insolubles %	0.02	0.04
Nontannin %	1.45	0.67
Tannin %	1.74	1.70
Purity Tan / SS X 100	54.6	71.7
Total Sugar %	0.8	0.06

The final member of the third group was isolated from a solution of canaigre liquor containing about 10 per cent of a synthetic tannin, Orotan,** resulting from an extraction experiment. After standing in the laboratory for several weeks this solution formed a light-colored sediment, which was sub-cultured to yield a yeast designated as Y 89. This culture has not yet been completely identified. It is probably a haploid form of a species closely related to, but not identical with *Hansenula anomala.* As shown in Table VII, canaigre liquor can be fermented by this culture without loss of tannin.

Further studies will be made with this organism since at present it looks very promising.

Discussion

On the basis of these and other tests, a plan shown in Figure 1 has been drawn up for utilizing the cycling process in a canaigre processing plant. Lack of suitable equipment has prevented our actually carrying out the complete process. It should be pointed out that our experimental conditions, e. g., incubation on a shaker, were not designed for maximum alcohol yield and, therefore, would hardly be retained in a plant process. No doubt the alcohol yields from hydrolyzates could be improved by anaerobic fermentation with mechanical agitation, although it is uncertain whether this would produce sufficient yeast cells for fermenting the liquors. Also there was little attempt made to determine the best method for hydrolysis of the residue from a cost and efficiency standpoint. Such studies would obviously be required for evaluation of the process as a commercial venture.

The spent residue from the extraction unit would be hydrolyzed by acid or enzymes. After any necessary neutralization, filtration, etc., the sugar-containing solution would be fermented continuously. The fermented hydrolyzate would be run through a continuous centrifuge or filter, the yeast cells going to the liquor fermenter and the beer to stills for recovery of the alcohol. The liquor fermenter would also run continously. The fermented liquor

**Mention of specific products in this paper is not to be construed as a recommendation or endorsement by the Department of Agriculture over similar products not mentioned.

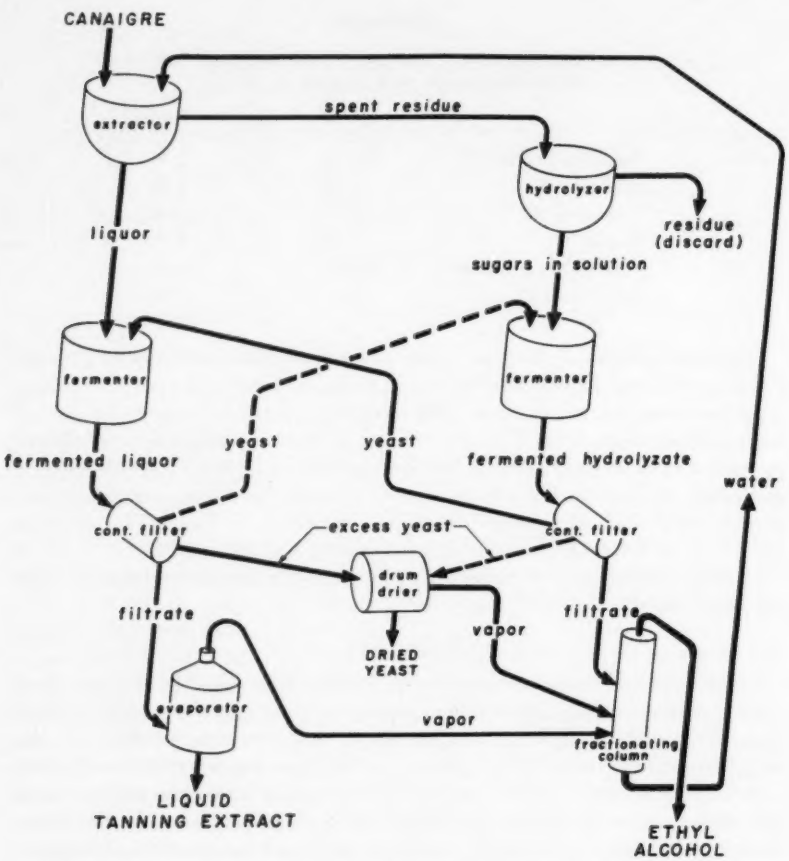

FIGURE 1.—Plan for utilizing cyclic yeast fermentation process in a canaigre extract plant.

would be centrifuged or filtered, the liquor going to an evaporator for production of liquid tanning extract and recovery of alcohol. The yeast cells, if needed, would be recycled to the hydrolyzate fermenter; any excess would be dried for feed as an additional by-product. If properly balanced, there appears to be no technological reason why this process should not be successful after the details have been worked out.

It would seem that enough alcohol might be produced to make its recovery economically feasible. It has been estimated * that a daily production of at

*Personal communication from P. Burke Jacobs, Industrial Specialist (alcohol) Northern Regional Research Laboratory, Peoria, Illinois.

least 2,000 gallons might be necessary in order to carry the essential plant labor and technological control without incurring too high an overhead charge per gallon of alcohol.

On the basis of figures which are admittedly rather tenuous, the production of 2,000 gallons of 95 per cent alcohol from the liquors and spent residues would require that from 90 to 100 tons of fresh roots be processed per day, yielding about 8–10 tons of dry extract containing 60 per cent tannin. Daily production of 10 tons of dry extract would amount to about 1,500 tons of 100 per cent tannin per year, based on 250 working days. This would constitute about 1¼ per cent of the total current domestic consumption of vegetable tannin, of which about 85 per cent is now being imported. If competitive price and quality of the extract were favorable, it is obvious that the market would readily support a number of plants such as the one proposed.

Summary

The action of various yeasts on canaigre liquors is described. Most of the yeasts tested were killed by the liquor. A few cultures were found which can tolerate the liquor and assimilate the sugars but they do not grow nor proliferate. Members of this group have been utilized in a process in which the yeast is first grown in a suitable medium such as hydrolyzed spent canaigre. The cells are then harvested and placed in the liquor to be fermented. After the sugars have been fermented the cycle is repeated. Alcohol may be recovered from both media. A plan is given for utilizing this process on a continuous basis.

A third group of yeasts consists of those that multiply in canaigre liquors and carry out normal metabolic processes. Three such species have been isolated. Two of these destroy tannin, but one can be grown in canaigre liquors without attacking tannin and is being investigated.

Acknowledgments

The authors wish to thank F. P. Luvisi and M. L. Happich for making the tannin and sugar analyses.

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50 Years of Progress in Chrome Tanning*

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It is a privilege to be asked to address this distinguished scientific audience. My subject, however, makes me appreciate my age when I realize that I have been a chrome tanner for almost fifty years following in the footsteps of my father, who was one of the first to produce two bath chrome tanned Horse Fronts and Veals in addition to the regular Vegetable and Dongola tannages made during the last quarter of the past century.

The two bath process, invented by Augustus Schultz, was the only chrome tannage then known. The one bath process was just being developed and was still in the experimental stage. By the way, I wonder how many of you know that Schultz, who was a chemist employed by Wm. Pickhardt & Kuttrof, now General Dyestuff Corporation, discovered the chrome tanning process while trying to find a way of producing leather which could be used to cover corset steels and prevent perspiration from rusting them. Therefore, it could be argued that if it had not been for women, who at that time were wearing corsets in order to make themselves more attractive, we would not today be chrome tanning skins and hides. Our industry should consider a monument to the corset, or possibly to Lillian Russell as portraying its advantages.

The two bath process as you all know consists of treating the skin with bichromate of soda or potash and acid, then reducing the chromic acid which is formed with sodium hyposulfite or sodium bisulfite. The resulting basic chrome sulfate tans the skin substance In the one bath process this reduction is made separately, and the skins are treated with the resulting basic chrome sulfate.

Perhaps I can make no better beginning to my history of chrome tanning than to go to Proctor's book "The Principles of Leather Manufacture" published just 50 years ago. Out of a total of 512 pages he devoted one chapter of 22 pages to chrome tanning, and two of those pages covered the iron tannage. He allots a part of two paragraphs to the chrome retanning of a vege-

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table tanned leather, and one short paragraph to the vegetable retanning of chrome tanned leather. The twenty pages on chrome tanning, however, contain what even today still are basic facts. While talking of Proctor it is of interest to note that recently I came across a copy of "Hide and Leather" issue of 1896 and found a series of articles by Proctor on "The Analysis of Used Liquors in Chrome Tannage", showing that seven years earlier, or fifty-seven years ago, he already had developed control methods and was urging their use in our industry.

Proctor's opinion of chrome tanning at that time was expressed as follows:— "Chrome tannages . . . have established their position in the manufacture of all sorts of light leathers, in competition with all the older methods, and (are) making a serious claim to a share in the production of belting and even sole leathers."

By 1903 the two bath process had been fully developed, and there has been practically no change or improvement during the past fifty years.

On the other hand, in 1903 the one bath process was just coming out of its experimental stage. Pickling before tanning seems to have been the exception rather than the rule.

Chrome chlorides and sulfates seem to have been used more or less interchangeably, although Proctor recognized that they behave differently. The effect of basicity was recognized, but there was considerable confusion between basicity and what we now know as pH. Thus Proctor refers to chrome chlorides as being less "basic" than sulfates when he meant that, for the same basicity, chrome chlorides have a lower pH value than the sulfates. The concept of pH was not brought out until several years later.

The idea of complex masked salts was still many years in the future, though Proctor clearly recognized the effect of organic acids when he wrote, "It is very probable that in the use of chroming baths containing organic acids, they have considerable influence on the tannage", and again, "the organic matters, and especially the organic acids which result from the oxidation of the sugar or glycerin, are not without influence on the tanning properties of the liquor." He also made the suggestion which has often been repeated that, "More satisfactory results in chrome tanning will be attained by the direct addition of known organic substances, to liquors of known constitution, than by the somewhat uncertain products of organic oxidation".

Proctor apparently did not consider it necessary, or even desirable, to add salt to chrome tanning liquor, but here he was at variance with the practice of the times, as shown by other publications.

It is well to consider the other process changes which are so closely associated with chrome tanning. Fifty years ago we tanners were soaking our skins from ten to twenty-four hours, dirty skins requiring a change of water. We knew that old soaks produced loose leather, and we believed that prolonged soaking in salt water gave us soft and empty leather. Most of us fleshed out

of soak and resoaked for 4-10 hours. Liming was done over a period of nine to ten days, starting in old, weak or used limes, and progressing through newer limes, finally ending in a fresh lime. Red arsenic was used in limes as it reduced difficulties with fine hair and gave a smoother, silkier grain.

Some tanners who were making side leather saved a few days by using sodium sulfide instead of arsenic, however calf tanners believed that this chemical was too strong to use on the delicate skin of a young calf, especially when they saw the strong swelling effect produced by its use. Not for a period of nearly 25 years did the fundamentals concerned become known, but today as a result of the fundamental work done by McLaughlin and his associates here in Cincinnati, we vary the soaking of our hides or skins to suit their cure and condition, as well as the kind of leather we desire. As the further result of work done at Cincinnati plus some of the early work at Lehigh University we are now able to handle intelligently the liming or unhairing operation through the proper use of sulfhydrates, amines, sulfides, etc., or combinations thereof, in 2 to 4 days, and to obtain better and more uniform results.

For nearly 80 years the calf tanners did head splitting or checking on a union splitter. This by the way was one of the hardest jobs in the tannery during that time, and it was not until 1945, when our Quartermaster Corps commandeered and brought over from Germany a band knife head splitting machine, which had been in use there for more than twenty years but was unknown over here, that this job became one of the easier tannery jobs.

In the later parts of the 19th century skins were unhaired and fleshed by machine, but all skins were hand beamed, an operation most of the present generation of tanners either do not know or of which they have only a hazy book knowledge. It consisted of placing the skin grain up over a rounded beam, sloping from almost waist high away from the operator to the ground, and scraping the grain surface with a dull knife, curved to the shape of the beam and held by one hand on each end, by pushing away and pressing down. This removed practically all fine hair as well as most of the scud or dirt from the hair follicles. Any spots of fine hair that remained were shaved off with a small sharp knife, an operation which today is done by few if any tanners, machines and process developments taking their place.

After unhairing and fleshing, the skins were held in cold water overnight. After a wash they were bran drenched, the forerunner of modern bating—an operation with which most of the present generation also is not acquainted. They only know of bran from radio advertising as a breakfast or health food. Fifty years ago we used it to remove the lime from the skins. Most of us did not know how it worked. It was about that time that some chemists told us that it produced lactic and other organic acids and pointed out how we could save time and do a better and cleaner job by using lactic acid. Quite a few of us, especially those who were making the finer leathers, used dog manure in place of the bran drench—tanners of goat, glove leather, etc., used bird

manure. We knew that it gave us a silkier and finer grain. At about this time "Rohm" in Germany and "Wood" in England were developing proof that it was the action of enzymes present in the manure that produced the grain effect we wanted. This resulted in the production and use of prepared bates and deliming agents—such as oropon, etc., which replaced the bran drench, lactic acid, and manures—saving time, eliminating odors, and helping to standardize our leather. After the manure bate we treated the skins with a dilute lactic acid bath to neutralize the alkalinity, and then scudded or slated them. This slating operation was similar to beaming or fine hairing, deriving its name from the fact that a tool made from soapstone was used instead of a steel knife. The skins were then rinsed, horsed up, allowed to drain, and weighed for tanning. You can see that in the period covered by this paper there has been great progress in the beam house process with attendant influence on the character of the product, i. e., from a general belief in the old days of weak and old, long liming and relatively little enzyme action during the liming. Today it is almost as general a practice to use a short, fresh and sharp lime, with a very much stronger enzyme treatment during the deliming.

The necessity of avoiding too rapid tannage, especially of the grain, was recognized, but the means for accomplishing this end were unnecessarily cumbersome and represented a carry over from vegetable tannage. It was common practice to pre-tan with aluminum by giving an alum pickle. Tanning was started in weak, previously used liquors, that were gradually strengthened, or the stock was moved from weak to stronger liquors. Apparently tanning was done in paddles or pits; even suspenders are mentioned. Drum tannage is mentioned but not advocated except for hastening the tannage. I remember that the normal time required for the chrome tanning of calf skins usually extended over some days.

Thorough washing usually before shaving, and thorough neutralizing before drying and fat liquoring were emphasized. Neutralization was apparently carried further than today, for I can dimly recall that neutralizing was carried to the point where the skins were no longer acid to litmus, using as much as 3 per cent of borax on the wet pelt weight. However, we did recognize the danger of over neutralizing.

Fatliquoring appears to have been done, as a rule, with soap-oil emulsions. It is evident from Proctor's comments that this operation frequently gave trouble, and that poor fatliquoring often resulted in finishing difficulties.

Very much of the same picture as to the state of chrome tanning is given in "Modern American Tanning", Volume I, issued by Jacobsen Publishing Co. (Hide and Leather Trade Paper of Chicago in 1902). It consists of a collection of articles that had been published in "Hide and Leather" and been written by well known tannery foremen, superintendents, and chemists. It includes a copy of the original chrome tannage patent of Augustus Schultz dated January 8, 1884. It had been filed May 31, 1883.

In the preface it states:—

"The introduction of the chrome or mineral tanning process has revolutionized the upper leather industry, and the modern process of wheel stuffing has almost completely superceded the old fashioned system of hand stuffing in the production of harness leather.

"The finishing of calfskins on the grain side has been so successful that the old system of currying the skins and finishing them with wax on the flesh side has been almost entirely discontinued."

The preface continues, stating:—"In recent years the manufacture of leather has been carried to a high place of efficiency and economy . . imports of leather have been reduced to a minimum and an immense export outlet has been opened for American leather and leather goods. This gratifying industrial victory was only made possible by the enterprise, ability, and industry of the American tanner, chemist, and mechanician."

The book also states:—"No leather introduced by the American tanners during the last few years has given more satisfaction as far as looks and wearing qualities are concerned than chrome tanned calfskins."

Fifty years have passed, and research both basic and practical done here at the Tanners' Council Research Laboratory at Cincinnati, as well as at Lehigh University and private industrial laboratories, has brought the chrome tanning process to still greater importance in the tanning industry. The stuffing process has been appropriated by the tanners of military shoe upper leather, work shoe leather, as well as mechanical leather—all tanned principally by chrome. Look at your and "My Lady's" feet and you will see the result of chemical and technological progress in the tanning and finishing of chrome calf leather for shoe uppers—beauty and service not dreamed of in 1902.

During the past fifty years the practical development of our industry has been greatly helped by the contributions of chemists such as Proctor, Wood, Rohm, Stiasny, McLaughlin, Theis, Kuentzel, Gustavson, Otto, etc., etc., some of them still active, and if I should summarize all contributions, I would say they have contributed to a better understanding of the process, resulting in considerable simplification, better control, increased knowledge of the action of masking agents, and greater uniformity of the finished product.

Fifty years ago practically all of us controlled our process by using our tongues as our pH meter. We tasted the skins and liquors at practically every point. Some of us who were a little more advanced also used litmus paper but only to confirm our taste. We used the boil test by observing when and how the leather shrank. The process required from 48 to 120 hours for calfskins.

Today we do it in 4 to 20 hours which allows us little time to correct errors. We, therefore, must prevent them by close chemical control at all times. We know something of the effect of the various neutral salts, and we use this

knowledge to produce desired modifications in the properties and appearance of our leathers.

We formerly dyed black leather by means of a large percentage of hematine or logwood topped with nigrosine. Now we use a small amount of logwood and dye with direct dyes containing, if desired, a small amount of acid dye. Our colors were formerly all dyed with basic dyes. Today it is either direct or acid dyes, though recently some of the newly developed basic dyes are proving of interest. We also use so-called syntans to obtain desirable grain effects, either a glassy smoothness or a pebbled shrinkage, depending on the type used. Whereas fifty years ago we used a fig soap and neatsfoot oil emulsion for fat liquoring, we have now changed to a sulfated oil and raw oil emulsion, and today some tanners have returned to an egg oil and neatsfoot or cod oil emulsion which does not contain any sulfonic groups therefore produces tighter flanks by not affecting the chrome complex. It is only within the past year that science has perfected a process for manufacturing egg oil without altering its lecithin and cholesterol content thus enabling the tanner to return to the type of leather fibre lubrication that produced the best leather for his ancestors even in the time of the Pharaohs.

We used to hang our leather in lofts for the first drying, then process it by conditioning, staking, and nailing it on frames for final drying before the finishing operations. Today a very large percentage is pasted with the grain against a glass or porcelain coated plate, passing through a continuous dryer and coming out dry in 3 to 4 hours, then stripped from the plate, washed clean of paste ready for the finishing operations. Other leathers which formerly were nailed on boards to dry are now toggled on to frames and passed through a dryer requiring 30–40 minutes instead of 8–10 hours.

There have also been great developments in the finishing of upper leathers. It is true there has been no change in the bower jack used for glazing or burnishing, but by means of pigments, dye lakes, and improvements in coating solutions a range and uniformity of colors is obtained undreamed of years ago. I remember when a tanner's production consisted of 80 per cent black and 20 per cent brown, then tan came in followed by gray; but still well over 65 per cent was black. Now look at the range of shades—our stylists dream of new ones every night, and we produce them or at least come reasonably close to their dreams. Coating solutions that were formerly applied by hand are now sprayed or machine applied and dried within minutes instead of overnight. We have developed synthetic resin coatings so that all except the higher grade upper leathers are now no longer glazed but are only ironed and smooth plated.

Looking into the future, chemistry and engineering will give still better control over variants and still further reduce the time between putting a skin into soak and measuring it when finished, ready to go to the shoe manufacturer; as for example the recent developments in solvent tannages either

resin, vegetable, chrome or combinations thereof, where the penetration is a matter of minutes, fixation requiring hours, and solvent costs and recovery apparently the only details still to be worked out. We will produce leathers that will wear better and be more beautiful than we dream of today as the result of basic and practical research in the field of tanning, dyeing and finishing which is now being carried on by many of you here today, including the laboratory here at Cincinnati.

Leather Finishing

SOME NOTES ON ITS HISTORY, PROGRESS AND IMPORTANCE*

By A. J. PILAR
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The art of leather manufacturing is an ancient one and therefore the complete story of finishing shoe leather would be very long. The purpose of this paper is to cover leather finishing over the last 50 years; however, to give a clear picture of this progress it is necessary to go back to the late 1800's and cover advancements made at that time. In the Sixties some of the first leather finishing machinery was developed for vegetable tanned leather and used in both wet and dry departments. Following this there was the transition period from slow vegetable to rapid chrome tannages.

Shoe leather made in the early 1800's was vegetable tanned and most of the work was accomplished by hard hand labor. Not until approximately 1865 did the tanners begin to use labor-saving devices. The first important ones were the scouring and setting out machines. These machines were the greatest asset to the tanner since they removed the "bloom", softened the leather, and smoothed the grain. Due to the inventions of Lockwood [1], Holmes [2], Fitzhenry [3], and Burdon [4], the production of leather was at once increased and the cost lowered.

The scoured leather was hung up to harden and then taken down and slightly dampened to temper a few days. It was now ready for stuffing. This operation was important in the making of strong, flexible, water resistant, and smooth grain leather. One of the successful stuffing compounds was an emulsion patented by Dr. Theodore D. Williams [5] in 1870. Neatsfoot oil, glycerine and tallow were emulsified with "glycerol of egg," which was composed of five parts of whole shelled eggs and four parts of glycerol. The compound was applied by hand to the flesh side of the leather, which was hung up to dry slowly.

After it was taken down it was set out on the Fitzhenry machine and then whitened by hand-slicking or the Enos & Clement [6] machine built in 1881.

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This operation removed the crusty hard grain of the leather so that it could be flexed without the grain checking. Whitening also removed dirt and grease from the hot-wheel stuffed leather.

"Stoning out" was next. In this procedure the leather surface was rubbed with a stone held in the jaw of a machine very much like a rolling jack, which helped to lay out the leather and produce a smoother grain.

If, after stoning out, the leather required softening, it was arm boarded. The first machine for boarding and graining was invented in 1871 by O. Coogen [7], in Pittsfield, Mass., and then later improved by G. W. Baker of Wilmington, Del. At this point the leather was ready for blackening.

Heavy shoe leather was finished by hand and light leathers by machine. Between 1880 and 1883 the F. H. Batchelder [8] blacking, coloring, and dressing machine was invented. To this day there has not been any great improvement made over this first seasoning machine. The grain of the leather was blackened with a mixture of lamp black, oil, and natural water-soluble gums. Naturally, this coating smutted and cracked. A special department was set up where the workers, called "rubbers" or "smutters," rubbed the grain of the fine quality shoe leathers with carpeting and fine cloths. This rubbing action caused the black film, called "skin," to loll up into little crumblike particles which were later swept off by brushing. This finished leather did not smut or drycrock and was sold at much higher prices. The Charles B. Bryant [9] machine, of the firm of Bryant & King, well-known Woburn, Mass., calf skin tanners, was introduced to replace the rubbers' and smutters' hand work. It was a revolving drum machine with alternating rows of bristle brushes and Brussels carpeting, and was a great help in reducing labor and speeding production.

Sometimes the leather was finished on the flesh side. In 1846 J. Hayward [10] made a flesh finish compound of potash soap with the addition of lamp black and menhaden oil. In 1868 Shaw [11] made a dressing of logwood extract, borax, gum shellac, bichromate of potash, and ammonia. He boiled the materials together, cooled them, skimmed off the skin, and strained the solution. This was a fine blue-black finish. In 1869 Joel Putnam [12] finished the inner side of bark splits with a grain renewer. It was made of hide glue, boiled linseed oil, vermilion or suitable pigment. It was applied to the split by brush, dried and glassed, then boarded.

The early practice of finishing vegetable side leather for shoes was to have the sides stuffed and set out by machine, whitened, stoned, and if firm, softened by boarding. The sides were then taken to the blacking loft and the flesh was colored with either lamp black and oil or soap. When the soap blacking was used, oil was applied freely over it in order to set the color. Soap blacking filled the flesh and made a level appearance. The blacking crocked off and the free alkali in the soap caused the leather to have a harsh feel upon aging. After the first blacking was applied, the leather was aired off and then glassed, tempered, and glassed again. The last operation was to

apply a thin coating of paste. The paste was made of two pounds of boiled hard soap, one pail of flour, one piece of tallow the size of an egg, and a small piece of wax, usually bees or a substitute Jap wax. The paste filled up the grain and made the stock "fine." The pasted leather was dried and glassed "in paste." Now the flesh had to be sized by hand sponging. A mixture of four ounces hide glue, warm water, tallow, and more warm water was used. The leather was hung up to dry, measured, marked and bundled for shipping.

Corrected sides were finished as follows: They were hand-stuffed on the table, hung up, set out, slicker buffed, and trimmed. Staining was done with logwood extract, and while slightly damp was struck with ironrust and copperas. This coloring operation was repeated a second time, after which the skins were smutted on the table, glassed, and horsed up. The grains were sponged with flour paste, dried and softened by boarding with flesh up. The flesh side was then cleaned by hand slicking and the grain was glassed. A clear tragacanth solution was applied on the grain over the flour paste and the skins were hung up and dried.

Waxed calf leather was finished by the following method: After stuffing the grain leather with fish oils, paraffin, oleine and rosin, it was set out, taken to the finishing room and blackened with logwood and struck with copperas, glassed, boarded, polished, oiled off with fish oil and paraffin. The so-called "straight grains" were jacked with a different roller and boarded in only one direction.

Morocco leather was a soft, pliable material, largely employed in the manufacturing of the uppers of ladies' and children's shoes and some gentlemen's low cut shoes, as well as bindings for books, linings for bags, toilet cases, pocketbooks, etc. In those days the finer grades of Morocco were made from goat and kid skins; the poorer from sheep or split calf skins. The trade names were many, but the outstanding ones were: imitation French kid, brushed kid, glazed kid, and straight grained goat. This vegetable tanned leather was made either by retanning imported East India tanned stock or tanning the imported raw stock. Morocco leather was stained by first applying a mordant, then the vegetable dye with a fine, soft brush, and then striking the color with a metal salt like copperas. Light brown shades were made with extracts or logwood, Brazil wood, fustic chips, and the addition of a small amount of alum. Bottle green was made with fustic, logwood, and copper sulfate.

In order to finish imitation french kid, the grain on kid skins was stained with logwood, Chinese nut galls, and iron liquor. The skins were then hung up to air dry, blackened again, omitting the nut galls, and adding a small amount of bichromate of potash, put on hooks with the blackened side inward until the color set, and then taken down and a solution of milk and water was applied to the grain. This leather was glazed by machine. After glazing it was softened with a hand-softening slicker. The grain was oiled off with the best grade of raw sperm oil, which was applied with a roll of soft

flannel cloth. It was glazed two or three times and oiled off twice. After the final oiling off it was glazed again.

The glazing machines were, without a doubt, responsible for making goat, kid, and calf upper shoe leather, and, in the opinion of the high grade light upper leather manufacturers today, it is almost impossible to produce this type of leather without a glazing machine. The machines were improved rapidly because of the scarcity of labor in the Northern States, due to the war of the Rebellion, and also the enormous demand for leather. In 1860 Martin [13] invented the slanting bed machine, next came Friend's [14] wooden frame, and in 1868 Hildreth [15] devised an improvement to get uniform pressure. In 1876 David Knox [16] received a diploma at the Philadelphia Exposition for greatest improvement and simplification in the glazing machine. Later, Bowers of Wilmington, Del., made the final improvement—a rocker pressure equalizer. The pressure and shock on the skin was not only cushioned by the bed yielding under the skin, but the arm holding the agate or glass was attached to this new wooden rocker arrangement, which also equalized the pressure from the top of the machine. This machine has never been changed. G. W. Baker [17] perfected the side glazer. His machine was called the Baker Pony Glazing Machine, or the Baker's Improved No. 4 Glazing Machine. The side machines were used extensively for final glazing, and the Bowers for first way glazing when great pressure was desired.

Brushed kid skins were given a logwood type blue-black stain and then dried. They were then coated with a finish of oxblood, iron, and vinegar black, applied with a roll of flannel. They were then wet over with a gum tragacanth solution and brushed with a very soft brush known as a "kid brush". After brushing, they were hung in the loft to dry and back-boarded to take out the curl and soften. They were then glassed, rolled with a steel rolling jack, scraped on the flesh side, and the grain oiled off with sperm oil.

Some examples of finishes patented in the Seventies follow: Mr. Hugh Smith [18] of Newark, N. J., invented a coating to make a new grain on buffs and splits. It was composed of glue, water, starch, or flour, aniline coloring, glycerine or fat, and pigments to suit. His second process was making leather water resistant and flexible with gum shellac, alcohol, India rubber or equivalent gum, naptha, glycerine, and dye thinned with wood alcohol.

In 1871, Franklin S. Merritt [19] of Boston, Mass., made imitation alligator leather. He tanned leather for japanning purposes, wet it, passed it between two rolls, one of which was engraved with an alligator design, boarded the leather lightly and dried. Then he took Xylonite sheets and stuck them to the leather with Collodion, passed the leather through smooth rollers and it was finished. Mr. Merritt not only had the idea of making embossed grains, but also the first nitrocellulose plastic coated leathers.

A Mr. T. May [20] made a finish of rye paste, gum tragacanth, American isinglass, oxalic acid, gum gamboge, water, and pipeclay. It had to be aged

seven days before using. It filled and covered, dried hard and bright, and was used mostly for sole leather bottoms.

In 1875 Mr. E. Fennessy[21] of Massachusetts made a bronze kid finish. The method for making simulated gold leather about this time was first to tack the wet leather and leave on frames. Next the grain was sized with isinglass solution and dried. A coat of egg whites was then applied as an adhesive for silver foil, which was pressed on with cotton. When dry, this silver-coated leather was given a coating of clear, high grade, golden-colored varnish.

Although a number of people had been working with chromium chemicals in the preparation of leather prior to 1884, Augustus Schultz[22] made the first important advance in practical chrome tanning. He was not a tanner but a dyestuff chemist working at that time for a New York aniline firm. The tannage was developed while he was trying to produce a special type of leather for a friend who wished to use it to cover corset steels, which were rusted by the other tannages. It was in the Nineties when Martin & Dennis[23] made the one-bath tan of basic chromium sulfate. In 1902, the writer received a patent for his development of a special one-bath tan[24] using the same ingredients as Schultz used in his two-bath process. The first chrome tanned leathers which were produced in large quantities were the light leathers, goat and kid. A few well-known tanners making chrome kid leather in approximately 1882 were Robert H. Foerderer, J. P. Mathews, both of Philadelphia, Pa., and F. J. Blatz & Bros.[25] of Elizabeth, N. J. Tanners saw the advantage of the chrome process and rapidly accepted it. This new method of tanning caused a great change in finishing leather.

The chrome tanners' production ranged from 1800 to 40,000 glazed kid and goat skins a day. Nearly all the leather was put into blacks. They used logwood, nigrosine, gelatine, alcohol, glycerine, carbolic acid, ammonia, a little chrome, and fresh red defibrinated oxblood. There has been little change to date, with the exception of the addition of a small quantity of good black pigment to fill up the damaged grain and darken the gray or silvery effect of the large hair cells. The finishers had their troubles making uniform leather because the skins were not properly fatliquored for finishing. Vegetable tanned leathers would absorb a large quantity of the oil and grease but the chrome leathers absorbed very little and the chrome salts would react with the fatliquor to make insoluble metallic soaps which could not be easily washed off, and the finishes would not penetrate. Sometimes the skin had to be degreased and most of the time cleared with special washes, such as barberry juices, oxalic, lactic, or acetic acids. After the first glazing the skins were cleared again.

A major advance in finishing came with the solution of the fatliquoring problem. When this problem was licked, they still had to contend with the sulphur spew. It bleached the colors and caused a dry haze in blotches over the skins. In the two-bath process the hypo bath was not kept cold enough

and very fine particles of sulphur, known as "flowers of sulphur," were produced. The fatliquor would migrate to the surface of the leather carrying the sulphur with it. Shoe men became so accustomed to seeing the "bloom" that when customers complained they were told that it was impossible to manufacture fine leather without it.

Chrome leathers were wet tacked and while on the boards the grain was sponged off with glycerine and water, then lightly oiled off on the grain with the best grade of sperm or neatsfoot oil. They were then taken to the drying loft and dried completely. After they were taken off the boards, they were put into sawdust, staked on the Slocomb, sammied, soft boarded, and a thin coat of flaxseed solution was applied to the grain, and dried. The skins were seasoned with a solution of egg albumen, milk, dye, phenol, and water. After drying they were glazed twice around, reseasoned with the above solution, reduced fifty per cent with water, and dried. A light perching was next, followed by boarding from the neck to the butt to get a straight grain, and rubbing off with raw linseed oil.

As the technology of chrome tanning advanced, so did that of finishing. Glazed kid and calf were no longer made only in black and a few browns, but they were being made in many bright colors with German aniline dyes. This aniline leather was beautiful when high grade raw stock was used. The only drawback was that soft skins were light, and hard grain skins dark. Often a drum of finished skins when sorted for color would yield a dozen shades. The imperfections were obvious. The cutting value was very low. Machine buffed sides were used for work shoes and boots because the leather was serviceable, but the appearance was far from dressy. Glazed leather and corrected sides had to be finished a new way to reduce the number of grades. This was accomplished by the use of embossing and smooth plating machines and concentrating on the development of pigments finishes. The tanners bought their colors ground in water (distemper) from the paint industry and made their own finishes. Burnt umber, sienna, ochre, Spanish oxides, lamp blacks, clays and some special pigments were used. The same binders, waxes, soaps, and dyes were used with the addition of a new plasticizer—sulfonated castor oil. These finishes frequently dusted off, settled out hard on the bottom of the container, became thick, and decomposed; however, tanners who realized the great covering power of pigments would not give up their use. About 1910 the first leather pigment finish manufacturers started in business. These companies spent their full time on making finishes which they sold in carload lots. Other interested parties jumped on the band wagon and started new companies. More finish producers brought more ideas and more service to the tanners as a result of this healthy competition.

During this era, nitrocellulose, oil, paint and varnish manufacturers were also experiencing a boom in the demand for their products. Pigments were

needed for their coatings and the chemical companies here and abroad quickly tried to fill the demand. They made chrome colors, iron blues, paratoners, special oxides of iron, lithopone, titanium dioxide, lithol toners, toners made by precipitating basic dyes with quebracho, and some special colors with tannic acid or other selected metallic salts. The best light, heat, and alkali-resistant colors were made from the late Thirties to Forties. They were the cadmiums, indanthrenes, phthalocyanines, special tungstated and molybdated pigments.

In about 1920 casein was featured as the major binder in leather finishes and has played an important part ever since. It was used with glue, gelatine, blood, and all the other film-forming materials mentioned.

Hugh Smith[17], as stated previously, used India rubber and Merritt[19] Xylonite with Collodion in the Seventies, but not until the First World War was the use of solvent type coatings really applied to leather in large quantities. Nitrated gun cotton was war surplus and it was a drug on the market. Tanners and textile men took this material, called "spaghetti," dissolved it in acetone, amyl acetate (banana oil), and wood alcohol. The thinner was benzene or some refined petroleum. The plasticizers were blown rape seed, blown castor, raw castor oil, and tricresyl phosphate. Colors were ground in oil and then added to the clear lacquers. They were applied by hand brushing at first, and then later by spraying. They were air dried. These coatings served their purpose but did not age well. Today we have nitrocellulose coatings which will last almost as long as the material to which they are applied. Some other solvent type coatings were developed in this order:

1920, Bakelite; 1927, Vinylite; 1930, Alkyds; 1935, Polystyrene and Urea Formaldehyde; 1937, Methacrylics and Acrylics and Nylon.

About 1940 lacquers which were emulsified with water were first being used to coat leather. They could be diluted with water instead of thinner, and were less hazardous. Water, dyes, binders, and specially dispersed pigments could be mixed with them. The material gave good levelness, was water resistant, and flexible. The emulsions found their place in the glove, coat, garment and novelty field. Later they were used to make aniline side leather. Although these materials have been made for over 15 years, there is still much to be learned to make them absolutely fool proof.

In 1936 a new type of resin in the form of latex or milk-like material was introduced to the textile, paper, and leather industries. It was the Vinyl group: Polyvinyl Acetate, Polyvinyl Chloride, Acrylic and Methacrylic Polymers and copolymers polymerized in emulsion or pearl form. These latices were, after slight adjustment, mixed with the regular finishes and produced tough, water resistant, uniform, and flexible coatings. They also made it possible to use a water base finish for a lacquer top coat. One well-known company has made acrylic latex resin finishes since the middle Thirties.

The Second World War came along and the supply of natural rubber was cut off, so the Government set up a Rubber Control Board to allocate this critical material. They also invested large amounts of money in research, development, raw materials, and production facilities and furthermore saw to it that industry received any help it required. The results were fantastic. The synthetic rubber production program taught our higher polymer chemist more in five years than was known by anyone before. The leather finish manufacturers who were on their toes also learned about polymers and applied the knowledge to their business.

The success of the leather industry will be based largely on the proper utilization of the available finishes. High grade leather with few imperfections should be coated with a minimum amount of finish in order to retain the natural beauty and texture of the leather. In low grade leather, where it is necessary to use large quantities of finish to construct a new grain, the proper use and application of synthetic resin finishes is imperative. If the tanner takes advantage of the rapid development of synthetic resins, which can be used to impregnate as well as coat, specialty leathers can be produced which will have improved resistance to abrasion, chemicals, solvents, etc., and will definitely have great advantages over the leather substitutes. By recognizing these facts, we will be prepared to meet the challenge of the next fifty years of leather finishing.

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BOOK NOTICES

Skin and Leather Defects. By Fritz Stather. 2nd Edition. Published by Springer-Verlag, Vienna, 1952. 178 + VII pages, 97 illustrations. Cloth, $7.15. The 1st Edition of this work appeared in 1934, and was reviewed in *This Journal* **30,** 128 (1935). The scope of the new edition is the same, but the arrangement has been changed, and not for the better. Instead of discussing defects by classes, the present edition lists all the defects in alphabetical order, beginning with "abfärben" (crocking) and ending with "zwickenechtheit, ungenügende" (inadequate fastness to pinching). This artificial sequence makes it hard for anyone who does not think in German to find what he is looking for, without scanning the whole table of contents. The nature, cause, and (sometimes) the cure of each defect is covered in varying detail,—from a few lines to several pages. The illustrations, typography, and paper are excellent, and copious literature citations are given. The first edition is said to have covered 100 defects. This one lists about 150, which some will take as additional proof, if any were needed, that the leather industry is going from bad to worse. This book should be well worth its price to anyone who can read German. H. B. M.

Gerbereichemie und Gerbereitechnologie. By Fritz Stather. 2nd Edition. Published by Akademie-Verlag, Berlin, 1951. 722 + XX pages, 195 illustrations. Cloth, 58 DM. The 1st Edition which appeared in 1948, and was reviewed in *This Journal* **45,** 762 (1950), was so well received that republication was necessary within 3 years. The author has taken advantage of this happy circumstance to bring the work up to date and correct the inevitable errors of the initial publication. Most of the chapters have been rewritten completely; more and better illustration have been supplied; but the scope of the work remains the same, and the new edition actually contains fewer pages than the old. As before, this book is a condensed account of both the science and the art of leather-making, plus 56 pages devoted to furs. To find a book of the same broad coverage in English, one would have to go back a generation to the last edition of Procters' Principles of Leather Manufacture. If the reviewer has a criticism, it is that coverage of non-German leather literature since 1945 appears to be rather spotty. Nevertheless this book is highly recommended to every leather chemist and technologist who is fortunate enough to read German.
—B. B. M.

ABSTRACTS

Drying Problems in the Leather Industry. By Willy Sell. *Oesterreichische Leder-Zeitung* **8,** 100 and 150 (1953). Drying problems in the leather industry are discussed and

processes and equipment are described and illustrated. Curves show relations between leather moisture, drying time, air velocity and air humidity. I. D. C.

The Quantitative Determination of Tannin with the Aid of Metal Salts. By A. Haglund. *Oesterreichische Leder-Zeitung* **8,** 119 (1953). For the quantitative determination of tanning by precipitating it with metal salts, the acetates of lead, zinc, cadmium, copper, nickel, cobalt, and manganese were investigated. The last three gave poorer results than the first four so no results with them are reported. It is impossible to use a direct method with weighing of the precipitate. An excess of reagent must be used to completely precipitate the tannin but this excess must then be removed before the detannized solution can be evaporated and dried. Removal of excess reagent by means of a cationic exchange resin was investigated. Soluble solids determination was made as usual, but it was necessary to pass the solution through the resin column because metal ions other than those of the reagent were removed. To remove tannin 100 cc. of tannin solution was treated with a metal acetate (copper, lead, zinc or cadmium), centrifuged a few minutes and filtered; 50 cc. of the filtrate was passed through a column of exchange resin, the column was washed with 150 cc. of distilled water and the solution and washings evaporated, dried and weighed. A hair drier was found useful in hastening evaporation of the 200 cc. of liquid. Theoretically the method appeared faultless but was inconvenient because of the large volumes for evaporation; also duplicates did not check well, possibly because all of the material was not easily washed from the resin. The following method was found to be satisfactory. Treat 100 cc. of the analytical strength solution with an excess, usually 5 cc., of 0.1 molar solution of the metal acetate; partially neutralize if necessary, centrifuge, and filter. Treat with hydrogen sulfide for a few minutes, filter, transfer 50 cc. to a beaker, warm to remove hydrogen sulfide, transfer to a weighing dish and determine dry matter as usual. The method is rapid; the nontannin solution is ready for evaporation in 30 minutes. Duplicates agree well. To obtain complete precipitation it is sometimes necessary to adjust the pH value. For this, pyridine was found to be especially suitable. It is a weak base so an excess does no harm and it is volatile and can be completely driven off. A pH of from 6.0 to 6.5 is found when 2 cc. of pyridine and 5 cc. of metal salt are added to 100 cc. of tannin solution. This pH is suitable for complete precipitation of tannin. For pH values above 7 ammonia was used. The results are shown in the following table, in which the letters have the following significances: F = French, D = German hide powder; a = 1 cc., b = 2 cc., c = 3 cc., d = 4 cc., e = 5 cc., and f = 6 cc. of pyridine; h = 1 cc., k = 2 cc., m = 3 cc., and n = 4 cc. of ammonia.

Kind of extract	Hide powder filter method F	Hide powder filter method D	Shake method D	Acetate of Cadmium	Acetate of Zinc	Acetate of Copper	Acetate of Lead	Lead, basic
Quebracho unsulfited	93.3	92.2	87.3	82.0b	88.9b 90.0e		93.7	
Quebracho sulfited	89.5	88.2	84.2	61.7b 86.4k	83.8b	82.4b	89.2	
Oak wood sound wood	62.6	61.6	57.4	56.8b	60.9b 58.0	63.0		
Oak wood rotten wood	63.8	——	56.8	44.1b	34.8 47.8b		71.7b	

Spruce bark	63.7	64.8	57.2	57.0k 59.0m	50.8b 62.2k 62.4m 56.1n		68.1a 67.9b 68.4d	
Myrobalans	76.5	75.8	66.4		55.1 66.1a 72.5c	71.4b	77.3	
Wattle bark	78.9	78.3	75.7	70.3e 76.3h 77.7k 77.4m	75.1b		83.0	
Sumac	66.3	66.7	57.6	55.3b	56.9	63.4	71.1	
Gambier	79.0	77.0	53.9		47.2b 51.7d 55.6f 72.9k		64.3	73.5
Sulfite waste	59.0	59.8	52.5			26.4b	52.7b	52.9

The author concludes that it appears possible to work out a satisfactory method for tannin using metal salts, and may be possible to distinguish true tannins, semitannins and non-tannins. Further work will be done with mixtures of different extracts and combinations with syntans. I. D. C.

Recent Developments in Methods for Tanning Sole Leather with Special Reference to the Use of Mimosa Extract. By G. H. W. Humphreys. *Oesterreichische Leder-Zeitung* **8,** 123 (1953). In Great Britain, in 1937, 36 per cent of the tannin used was from wattle bark or extract. In 1950, 61 per cent of the tannin was from wattle,—all extract. Wattle extract as delivered can be used for many types of leather but better results are obtained with warm liquors at pH 3.2-3.4. Flexible sole leather can be made with lightly acidified liquors. Acidification increases fixation of tannin and degree of tannage, decreases water solubles and water absorption but does not change yield. The kinds of acid that may be used for acidification were discussed recently in "Das Leder". The following practical example is given: To prepare 2000 kilograms (kg.) of liquid extract (22-23°Bé) run 1000 liters of water into an agitator and heat with steam. If a bleached extract is desired add 5 kg. of powdered sodium sulfite and 2.5 kg. of formic acid (80%) to the water. Add 1000 kg. of wattle extract; this should dissolve in 6-8 hours. The temperature should not exceed 90°C. To acidify to pH 3.5 add either (a) 8 kg. of 80 per cent formic acid or (b) 18-20 kg. of 75 per cent lactic acid or (c) 20 kg. of acetic acid plus 3-4 kg. of formic acid. To acidify to pH 3.2 add (a) 14-15 kg. of formic acid or (b) 32-35 kg. of lactic acid or (c) 20 kg. of acetic acid plus 10 kg. of formic acid. The liquor may be used in hot pits or for extracting in a drum. I. D. C.

Raw Hides. By Karl Gabriel. *Oesterreichische Leder-Zeitung* **8,** 128 (1953). A brief review of hide defects and of quality and grades of hides from some of the principal hide

markets of the world. Hides are sometimes sold on the salt-free basis or with a specified percentage of salt. During salting with good drainage a hide loses in 14 days 10-15 per cent in weight and on further storage 3-6 per cent. Frigorifico hides lose 18-25 per cent in weight during curing and 5-10 per cent more on the ship. On drying, hides lose 55-60 per cent of their green weight. White weight yields based on green or salted weights are as follows: Clean summer hides 88-95 per cent; frigorifico hides 100-115 per cent; dry Buenos Aires hides, summer kill 190-220 per cent, winter kill 180-210; Agra Kips 220-250 per cent. For European animals there is a difference between summer and winter kill (between short and long hair) of 3 to 5 per cent for animals kept in stalls, and of from 4 to 8 per cent for animals on pasture. I. D. C.

Problems in Dyeing and Finishing Glove Leather. By F. Krames. *Oesterreichische Leder-Zeitung*, **8**, 134 (1953). A detailed discussion of numerous phases of the dyeing and finishing processes for various kinds of glove leather. I. D. C.

The Influence of Tanning on the Combination of Dyes with Leather. By H. Loewe. *Oesterreichische Leder-Zeitung* **8**, 140 (1953). The work of Otto is reviewed (see *this Journal* **48**, 588). Dyes may be bound to collagen by ionic forces or by dipole forces through peptide groups. Ionic bonds are easily hydrolyzed, have low affinity and therefore promote penetration. Dipole bonds have high affinity and so promote surface dyeing. Heat weakens the first type and strengthens the second type of bond so that dyeing is done at the highest temperature that is safe for the leather. Positively charged chromium complexes block the carboxyl groups of the leather and decrease its affinity for cationic dyes but increase its affinity for anionic dyes. The stability of a complex depends on the metal ion radius and is therefore high for complexes of iron, chromium and aluminum which have small radii and low for cobalt and sodium which have large ion radii. The ligand (attached group) also influences stability. With acids, stability increases as the dissociation constant decreases so that formate forms less stable complexes than acetate, or sulfate than sulfite. Stability of complexes increases on aging. Tanning with cationic chromium tanning agents gives a positive charge to the collagen, increases its isoelectric point from 5 to 6 or 7 and increases the affinity for anionic dyes. Leather tanned with highly basic aluminum salts can be dyed to unusually clear, deep tones. Alum tanned leather has no greater affinity for dyes than untreated collagen. Vegetable tannins and syntans have weakly acid phenolic groups and also carboxyl groups. Althought ionic groups attract the tannin to the hide, the important tanning bonds are the hydrogen bonds to the peptide groups. Cationic dyes rapidly dye vegetable leather but only the surface; they have poor rub resistance and may bronze. The suitability of certain commercial dyes for various leathers is discussed. I. D. C.

The Technic of Dressing and Dyeing Furs with Processes and Products. By O. Markgraf. *Oesterreichische Leder-Zeitung* **8**, 145 (1953). Practical methods are given for tanning, fatliquoring and dyeing fur pelts. Tanning by the Leipzig method with an acid-salt mixture is good but the product is not water stable. Alum forms complexes with many substances but does not form inner complexes as does chromium. The complexes hydrolyze with alkali. Alum tanned furs are not entirely stable to water. Formaldehyde is of little value except as a pretannage for the above tannages. A chrome tanned pelt is too leathery and can not be pulled out as desired. Chrome also colors the hair slightly. Before

dyeing it is necessary to kill the fur and mordant it. Alkali is almost invariably used for killing. Mordants are metal salts. Chromium is used for light, dark, and black colors and for guard hairs. For dark and black colors copper or iron mordants or various mixtures of these with or without chromium are used. I. D. C.

Mode of Action and Possible Use of Petrik's Passive Paper in the Leather Industry. By Franz Kirnbauer. *Oesterreichische Leder-Zeitung* **8,** 154 (1953). Packing paper impregnated with passivating salts, that is with salts of alkaline earth metals, were much more effective in preventing rust on metal than wrapping paper saturated with oil, wax or similar materials. Passive paper prevents access of atmospheric oxygen and renders metal wrapped in it passive. I. D. C.

Acidity, the Principal Difference between Modern English and German Sole Leather Tanning Methods. By G. H. W. Humphreys. *Das Leder* **4,** 97 (1953). Sole leather has two principal functions—to wear as long as possible and to retard water penetration. In America considerable time and expense is devoted to finishing sole leather to improve its appearance and its workability in the shoe factory. Wear is emphasized in Germany and England but the former tans at pH 4-5 and England at 3-3.3. Formerly with weak liquors it was necessary to tan for a year or more but with stronger liquors and extracts the time can be greatly shortened. However the question then arises as to what acids to use and in what amounts. An investigation was made of several acids for acidifying mimosa (wattle) tan liquors. Ox hides were tanned 54 days in liquors increasing from 0.3°Bé to 13°Bé. Except for the control, the pH was kept at 3.8 until penetration was complete after which it was lowered first to 3.7 then to 3.6. After 9 days in 13° liquor the hides were put in a fresh 19° liquor adjusted to pH 3.4 for 10 days. The leather was then rinsed, oiled, and dried. The following table (first 4 columns) shows the kinds of acid used, the milliequivalents per liter required to bring the 6.7°Bé liquors to pH 3.8, the number of days required for tannin to penetrate through the hide and the degree of tannage of the leather. Unexpectedly, all leathers had nearly the same degree of tannage. The leathers were then dried and immersed for 4 days at 40°C in 19°Bé liquors adjusted to pH 3.2 (hot pit process). The last 3 columns of the table show the milliequivalents of acid required, the degree of tannage, and the percentage of water solubles removed at 45° from the leather.

Acid	Mg. eq. acid for 6.7°Bé liquor	Days to penetrate	Degree of tannage	Mg. eq. acid	Hot pit Degree of tannage	Water soluble 45°
Control	13	19	48	35	58	28.5
Sulfuric	23	19	47	85	61	28.1
Hydrochloric	25	21	47	88	66	24.2
Formic	53	23	47	205	70	24.2
Citric	125	22	47	280	102	15.0
Lactic	63	28	49	190	107	13.9
Acetic	333	33	57	1290	122	9.1

Very high degrees of tannage were found with citric, lactic, and acetic acids but the leathers were short fibered, brittle and not of commercial quality. Thus at the same pH values some leathers were damaged and others undamaged. The hot pit process at pH 3.0-3.3 is used commercially to produce good leather. The kind of acid mixture is therefore the dominating factor. There must be careful adjustment of weak and strong acids. Commercial liquors also contain salts which repress swelling. Few tanners know that wattle can be used to make as firm a leather as chestnut. A table is given showing the amounts of different acids required in a 19°Bé wattle liquor to reach various pH values down to 3.2. In England most tanners use from 1 to 1.5 per cent of formic acid or a mixture of acetic acid with either formic or lactic acid. Leather (A) drummed for 2 days in wattle extract at its natural pH value. Degrees of tannage based on extraction of solubles at 18° were 94.5 and 106.0 for leathers B and A respectively. The corresponding values based on extraction of water solubles at 45° were 80.1 and 88.2. Laboratory and practical experiments are cited to show that acid may either be taken up by leather from the tan liquor or be given off by leather to the liquor depending on equilibrium conditions. I. D. C.

Treatment and Disposal of Tannery Waste Waters. By H. Scholz. *Das Leder* **4**, 121 (1953). Formerly tanners were not inclined to let outsiders into the plant and the sanitary chemist was only told the amount of waste water and nothing of its nature. The treatment task, however, should begin in the plant and not at the sewer. Other lands, especially the U.S.A., have given more attention to this matter. There are three types of tanning processes to be considered: vegetable, mineral and oil. Standard methods may be used for suspended matter, oxygen requirement, and biochmical oxygen demand (BOD). Suspended matter can not be limited to 50 milligrams per liter as is done for municipal sewage; it will consist of proteins, phlobaphenes, precipitates of lime and protein, etc. The oxygen requirement will be influenced by protein, sulfides, bate, sulfite from the bleach, formic acid from the pickle, sulfited tannins, dyes, etc. Dissolved organic matter may be classified into the following 3 groups: (1) tannin from vegetable tanning characterized by a high BOD; it can be precipitated by mixing this waste with that from the beam house. (2) aniline dyes that have visible color as well as a high BOD, but that can be removed by absorption on flocculated material in mixed liquors. (3) protein and its decomposition products which have a high BOD, are colloidal, and are quite difficult to remove. In a fine leather tannery (R Ihm, Raunheim) the amounts of water used were as shown in the following table:

Operation	Liters of water per skin		
	sheep	goat	calf
Soak	15	12	36
Lime	15	15	36
Rinse	200	90	97
Bate	7	5	18
Tan	6	6	9
Rinse	7	7	8
Dye-fatliquor	50	65	86

Tannery waste can be divided into the following categories and approximate percentages, based on total volume: (1) 8 per cent of strongly alkaline lime liquor and solid lime heavily loaded with colloidal protein. (2) 30 per cent of other water from the beam house;

this is neutral or slightly alkaline, has a low BOD, and contains suspended matter that can be removed mechanically. (3) 3 per cent of acid tanning waste with inorganic salts, if from mineral tanning, or organic matter with high BOD, if from vegetable tanning. Acidity can be removed by mixing this waste with beam house waste. (4) 9 per cent waste from dyeing and fatliquoring, characterized by weak acidity and color. (5) 50 per cent wattle water from cooling, heating, offices, etc. It can be seen that 92 per cent of the waste is relatively harmless and needs no special chemical treatment. Wastes of types 2, 3 and 4 above can be mixed then allowed to clarify in the main settling basin. Type 5 waste can be discharged directly to the sewer. The waste lime liquor (type 1) must be neutralized; this can be done with an acid waste such as that from chrome tanning but is best done with flue gas. For example, this waste may be screened to remove large particles, fed to a settling tank, then to a tank where it is treated with flue gas, to a third tank where it is allowed to settle overnight and then is discharged to the main settling basin with wastes of types 2, 3 and 4. Preliminary instructions are given for tannery waste treatment and disposal. They were prepared by a commission on tannery wastes and are for discussion and criticism. I. D. C.

Methods for the Examination of Tannery Effluents. By H. Spahrkäs. *Das Leder* **4**, 129 (1953). Methods are given for pH, suspended matter, oxygen consumed from permanganate, biochemical oxygen demand and sulfides. These methods are the same in most respects as methods given in "Standard Methods for the Examination of Water and Sewage." I. D. C.

XXIII

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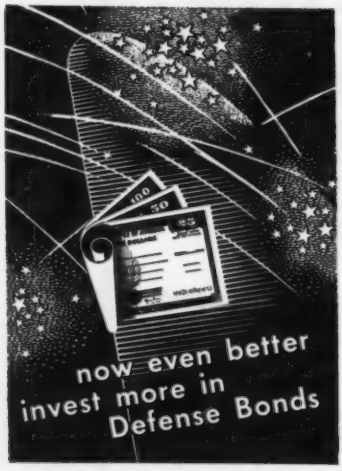

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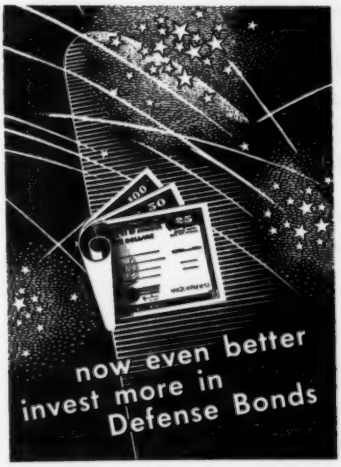

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